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THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

ORDINARY MEETING, DECEMBER 3, 1919.

HELD at the Chemical Society's Rooms, Burlington House. Dr. Samuel Rideal, President, in the chair.

A certificate was read for the first time in favour of Mr. William John Read, M.Sc., F.I.C.

Certificates were read for the second time in favour of Messrs. Percy Ewart Bowles, F.I.C., Ph.D. (Heidelberg), Charles Crowther, M.A. (Oxon), Ph.D. (Leipsic), John William Hawley, B.Sc. (Lond.), A.I.C., Ernest Joseph Lush, B.A. (Cantab.), A.I.C., H. Percy Marks, B.Sc., A.I.C., Robert Selby Morrell, M.A. (Cantab.), Ph.D. (Wurzburg), F.I.C.

The following were elected Members of the Society: Messrs. John Dalton and William Norman Leng.*

The following papers were read: "New Distillation Method for Detecting Adulteration in Butter and for Estimating Fats of the Coconut Group," by George Van B. Gilmour, B.Sc. (Lond.), A.R.C.Sc.I., A.I.C.; "A New Process for the Determination of Arsenic, with Notes on the Chemistry of the Marsh-Berzelius Process," by B. S. Evans, M.C., B.Sc., F.I.C.

The Council's nominations of Officers and Council for 1920 were read as follows:
President: Alfred Smetham.

Past-Presidents Serving on the Council: Messrs. Leonard Archbutt, Edward J. Bevan, A. Chaston Chapman, Bernard Dyer, Otto Hohner, Samuel Rideal, E. W. Voelcker, J. Augustus Voelcker.

Vice-Presidents: W. J. A. Butterfield, G. N. Huntly, G. R. Thompson.

Hon. Treasurer: Edward Hinks.

Acting Hon. Treasurer: E. W. Voelcker.

Hon. Secretaries: P. A. Ellis Richards, E. Richards Bolton.

Other Members of the Council: Messrs. F. W. F. Arnaud, W. Bacon, F. H. Carr, H. G. Colman, G. D. Elsdon, P. J. Fryer, R. Hellon, H. Hurst, C. A. Keane, S. E. Melling, G. W. Monier-Williams, W. Lincoln Sutton.

* Andrew More, F.I.C., was elected a member on November 5, 1919.

A NEW METHOD FOR DETECTING ADULTERATION IN BUTTER AND FOR ESTIMATING FATS OF THE COCONUT GROUP.

By GEORGE VAN B. GILMOUR, B.Sc. (LOND.), A.R.C.Sc.I., A.I.C.

(Read at the Meeting, December 3, 1919.)

THE chief difference between this method and those now in general use is in the treatment of the volatile acids. This method separates the volatile acids into two groups, depending on their solubility or insolubility in saturated brine.

Three numbers are obtained from which is deduced the genuineness of the fat under examination.

The test is carried out as follows:

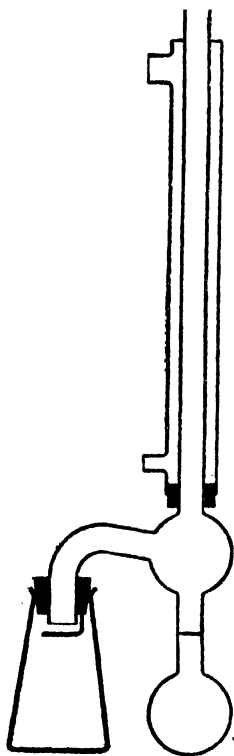
Twenty grms. of the clear filtered fat are saponified in a 300 c.c. resistance conical flask with 30 grms. of glycerol and 8 c.c. of 50 per cent. aqueous caustic potash; a few small pieces of porous porcelain are also added. The heating can be done over a naked Bunsen flame. The soap is made up to 200 c.c. with distilled water. To 50 c.c. are added 100 c.c. of a solution of sulphuric acid containing 12.5 grms. of concentrated acid per litre, and 0.1 gm. of pumice powder that has been sifted through butter muslin.

The distillation is carried out in the Blichfeldt distillation apparatus* shown in the diagram, which is calibrated to hold 100 grms. of water at 65° C., the source of heat being an electric heater regulated to distil to the mark in about twenty minutes. When the distillate has been collected, the apparatus is disconnected, and 0.5 c.c. of 1 per cent. phenolphthalein and $\frac{N}{10}$ NaOH (a few c.c. in excess of that which is necessary to neutralise all the acids in the distillate) are added through the condenser tube. The openings are corked and the contents well shaken. At the commencement of the shaking the cork is temporarily released to reduce the pressure inside. The contents are then removed to a 200 c.c. measuring flask and cooled to about 15° C. The excess of alkali is titrated with $\frac{N}{10}$ H₂SO₄, and the number of c.c. of $\frac{N}{10}$ NaOH, equivalent to the total volatile acids is obtained by difference: from this is subtracted 0.4 to allow for a blank, and the corrected figure is represented by T.

Up to this stage the procedure is identical with that described by Blichfeldt (*J. Soc. Chem. Ind.*, 1919, **38**, 150-152 π).

To the measuring flask containing the neutral soap is now added a volume of $\frac{N}{10}$ H₂SO₄ equal to that of $\frac{N}{10}$ NaOH required to neutralise the total volatile acids (T+0.4), and after this are added 61 grms. of pure dry sodium chloride (the salt used should be neutral to phenolphthalein, otherwise a slight correction will be necessary)

* The complete apparatus can be obtained from F. E. Becker and Co., Hutton Wall, London, E.C. 1.



The flask is corked and well shaken to dissolve the salt, the volume being made up to 200 c.c. The contents of the flask are next filtered, and 190 c.c. of the filtrate titrated with $\frac{N}{10}$ NaOH: no further indicator need be added, as phenolphthalein is already present.

The number of c.c. of $\frac{N}{10}$ NaOH required is multiplied by $\frac{20}{19}$ and from this is subtracted 0.4 for a blank. The number obtained represents the soluble volatile acids, and is indicated by S. T-S gives the number I, which represents the insoluble volatile acids. The following example shows how the results are tabulated.

$\frac{N}{10}$ NaOH added to the distillate	...	...	...	c.c.	26.0
$\frac{N}{10}$ H ₂ SO ₄ required to neutralise excess of alkali	...	...	...		4.4
					21.6
Deduction for blank experiment	...	...	...		0.4
$\frac{N}{10}$ NaOH required for total volatile acids (T)	...	...	...		21.2
$\frac{N}{10}$ H ₂ SO ₄ added to liberate volatile acids	...	...	...		21.6
$\frac{N}{10}$ NaOH required to neutralise volatile acids dissolved in 190 c.c. saturated brine	...	...	...		3.3
$\frac{N}{10}$ NaOH required to neutralise volatile acids dissolved in 200 c.c. saturated brine $3.3 \times \frac{20}{19}$	...	...	...		3.5
Deduction for blank experiment	...	...	...		0.4
					3.1
$\frac{N}{10}$ NaOH required for volatile acids soluble in saturated brine (S)	...	...	...		3.1
$\frac{N}{10}$ NaOH equivalent to volatile acids insoluble in saturated brine (T - S = I)	...	...	...		18.1

T = 21.2; S = 3.1; I = 18.1.

Saturated brine was adopted after experimenting with brines of different strength. The solubility of the fatty acids was found to decrease with increased concentration of brine.

Below are given figures illustrating this:

Grms. of NaCl in 200 c.c.	S for Butter Fat.	S for Coconut Fat.	S for Palm Kernel Fat.
61	22.5	1.6	1.1
40	24.6		
20	26.1	3.7	2.1

Results obtained by the above method for butter, coconut, palm kernel and babassu fats:

T represents total volatile acids.

S represents volatile acids soluble in saturated brine.

I represents volatile acids insoluble in saturated brine.

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Twelve Samples of Butter from Different Sources.

Sample.	T.	S.	I.
1.	27.2	20.4	6.8
2.	29.3	22.0	7.3
3.	26.3	20.3	6.0
4.	28.6	21.8	6.8
5.	30.1	22.8	7.3
6.	30.4	22.5	7.9
7.	29.4	22.0	7.4
8.	26.4	21.1	5.3
9.	31.0	22.5	8.5
10.	31.4	22.8	8.6
11.	32.2	23.3	8.9
12.	29.1	22.2	6.9

Six Different Samples of Coconut Fat.

1.	20.3	1.5	18.8
2.	19.7	1.6	18.1
3.	22.0	1.7	20.3
4.	21.0	1.5	19.5
5.	20.4	1.6	18.8
6.	21.3	1.7	19.6

Six Different Samples of Palm Kernel Fat.

1.	12.8	1.1	11.7
2.	13.1	1.2	11.9
3.	13.8	1.3	12.5
4.	12.4	1.0	11.4
5.	12.8	1.0	11.8
6.	12.0	1.0	11.0

A Sample of Babassu Fat gave the following figures

14.3	1.2	13.1
------	-----	------

Variations for butter, coconut and palm kernel fats:

<i>Butter Fat.</i>		<i>Coconut Fat.</i>		<i>Palm Kernel Fat.</i>	
T.	From 26.0 to 33.0	T.	From 19.5 to 22.5	T.	From 12.0 to 14.0
S.	20.0 „ 23.5	S.	1.3 „ 1.8	S.	1.0 „ 1.3
I.	5.0 „ 9.5	I.	18.0 „ 20.7	I.	11.0 „ 12.7

Below are given results for—

- (1) Mixtures of butter fat with coconut and palm kernel fats.
- (2) Pure fats used in (1).
- (3) Theoretical figures that the mixtures (1) should give calculated from (2).

	T.	S.	
(1) Butter fat + 5 per cent. coconut fat ...	28.3	20.0	8.3
" 10 " " " ...	27.8	18.8	9.0
" 20 " " " ...	26.9	17.0	9.9
" 5 per cent. palm kernel fat	27.9	20.2	7.7
" 10 " " " "	27.3	19.1	8.2
" 20 " " " "	25.7	16.8	8.9
(2) Butter fat ...	28.7	21.0	7.7
Coconut fat ...	21.0	1.5	19.5
Palm kernel fat ...	13.1	1.2	11.9
(3) Butter fat + 5 per cent. coconut fat ...	28.3	20.0	8.3
" 10 " " " ...	27.9	19.0	8.9
" 20 " " " ...	27.1	17.1	10.0
" 5 per cent. palm kernel fat	27.9	20.0	7.9
" 10 " " " "	27.1	19.0	8.1
" 20 " " " "	25.6	17.0	8.6

The following are results for—

(1) Mixture of arachis oil with coconut and palm kernel fats, and of arachis oil, butter and coconut fats.

(2) The pure fats used in (1).

(3) Theoretical figures that the mixtures (1) should give calculated from (2).

	T.	S.	I.
(1) 50 per cent. arachis oil + 50 per cent. coconut fat ...	11.1	1.3	9.8
50 " " " " palm kernel fat ...	8.0	0.8	7.2
70 " " " 30 per cent. coconut fat ...	7.8	1.0	6.8
70 " " " " palm kernel fat ...	4.7	0.7	4.0
30 per cent. coconut fat + 60 per cent. arachis oil + 10 per cent. butter fat ...	10.7	2.9	7.8
30 per cent. coconut fat + 65 per cent. arachis oil + 5 per cent. butter fat ...	8.7	1.8	6.9
(2) Butter fat ...	30.9	22.5	8.4
Coconut fat ...	22.0	1.7	20.3
Palm kernel fat ...	13.8	1.3	12.5
Arachis oil ...	1.0	0.3	0.7
(3) 50 per cent. arachis oil + 50 per cent. coconut fat ...	11.5	1.0	10.5
50 " " " " palm kernel fat ...	7.4	0.8	6.6
70 " " " 30 per cent. coconut fat ...	7.3	0.7	6.6
70 " " " " palm kernel fat ...	4.8	0.6	4.2
30 per cent. coconut fat + 60 per cent. arachis oil + 10 per cent. butter fat ...	10.3	2.9	7.4
30 per cent. coconut fat + 65 per cent. arachis oil + 5 per cent. butter fat ...	8.8	1.8	7.0

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Interpretation of the figures obtained by the method :

Genuine butters have been found to give figures which agree with one of the following conditions :

	Soluble.	Insoluble.
1.	20.0 to 21.0	Below 7 (usually 6.7)
2.	21.0 „ 22.5	„ 8.0
3.	22.5 „ 23.5	„ 9.5

When butter is adulterated with coconut or palm kernel fat the soluble volatile acids from either of them affect the soluble figure only very slightly—viz., by about 0.15 for each 10 per cent. of coconut fat, and 0.11 for each 10 per cent. of palm kernel fat present. The soluble figures for butters adulterated with these fats can thus be assumed to decrease approximately in proportion to the amount of either fat present.

The insoluble volatile acids from coconut and palm kernel fats affect the insoluble figure of a butter adulterated with these fats by about 1.9 for each 10 per cent. of coconut and 1.2 for each 10 per cent. of palm kernel fat present.

The insoluble figures for butters vary between 5 and 9.5, therefore on addition of coconut or palm kernel fat the butter portion of the insoluble figure will decrease by 0.5 to 0.9 for each 10 per cent. of these fats present, but, at the same time, the portion of the insoluble figure due to coconut or palm kernel fat will cause an increase respectively of 1.9 and 1.2. The result is that a butter adulterated with increasing quantities of these fats will give soluble figures that consistently fall, and, at the same time, insoluble figures that consistently rise. Butter adulterated with fats that do not give rise to volatile acids will give figures both soluble and insoluble reduced in proportion to the amount of adulteration.

The amount of adulteration is calculated in the following way :

Using average figures for pure butter, coconut, and palm kernel fats, the following three equations have been worked out, from which can be calculated directly the percentage of any one of the fats present in a mixture :

- (1) Per cent. butter fat = $4.67 S - 0.35 I = x$.
- (2) Per cent. coconut fat = $5 I - 0.38x$.
- (3) Per cent. palm kernel fat = $7.69 I - 0.59x$.

Should butter fat be absent from the mixture the second terms of the equations (2) and (3) vanish.

For differentiating between coconut and palm kernel fats see the paper by S. H. Blichfeldt (*loc. cit.*).

Distillation figures for mixtures of known composition showing results calculated from the above equations :

Actual Composition.				T.	S.	I.	Calculated from Equations.		
Butter Fat per Cent.	Coconut Fat per Cent.	Palm Kernel Fat per Cent.	Araohis Oil per Cent.				Butter Fat per Cent.	Coconut Fat per Cent.	Palm Kernel Fat per Cent.
10	90	—	—	21·2	3·1	18·1	8·1	87·4	—
95	—	5	—	27·9	20·2	7·7	91·6	—	5·2
5	30	—	65	8·7	1·8	6·9	6·0	32·2	—
10	30	—	60	10·3	2·9	7·4	10·9	31·8	—
—	—	50	50	8·0	0·8	7·2	—	—	55·4
50	5	—	45	15·7	10·7	5·0	48·2	6·7	—
40	15	—	45	14·8	8·6	6·2	38·0	16·6	—
10	60	—	30	15·9	3·0	12·9	9·5	60·9	—

There is considerable difference between the maximum and the minimum figures that can represent the total volatile acids in butter. The result of this is that a butter with a high figure can be adulterated with up to 10 per cent. of lard or other fat that does not give rise to volatile acids, without the figure being reduced below the lower limit. Such cases of adulteration cannot be detected by the distillation tests: if the butter is suspected, other tests must be applied.

Of the methods in use for detecting adulteration in butter and for estimating fats of the coconut group, those of Kirschner-Polenske and Blichfeldt are the most reliable. In the "Maypole" laboratory the latter method has been preferred. This new method has been tested against Blichfeldt's, and has been found simpler and somewhat more exact; a complete estimation can be made in about an hour.

It has been noticed that the ratio of soluble to insoluble silver salts in the Blichfeldt process is to some extent dependent on the quantity of fatty acids present. This might also be true in the Kirschner process. The fact that, by using saturated brine, the ratio of soluble to insoluble volatile acids is not affected by the quantity of the acids present, is a point very strongly in favour of the method.

THE LABORATORY,

MAYPOLE MARGARINE WORKS, LTD., SOUTHAL, MIDDLESEX.

DISCUSSION.

Mr. BOLTON remarked that he could not criticise the efficiency of the process without studying the figures which he had only just seen for the first time. He felt that, in order to judge the value of this new method, the figures should be tabulated side by side with those obtained by the standard Reichert-Meissel-Polenske-Kirschner method, in order to distinctly prove the advantages claimed. The standard Reichert-Meissel-Polenske-Kirschner method would detect and even estimate 1 per cent. of butter fat in the presence of coconut oil and palm-nut oil.

If the method now put forward would do this more accurately or more rapidly then it would be for the analyst to decide if it was worth while to scrap the great number of published figures for the standard method, and to obtain fresh standards for the author's method.

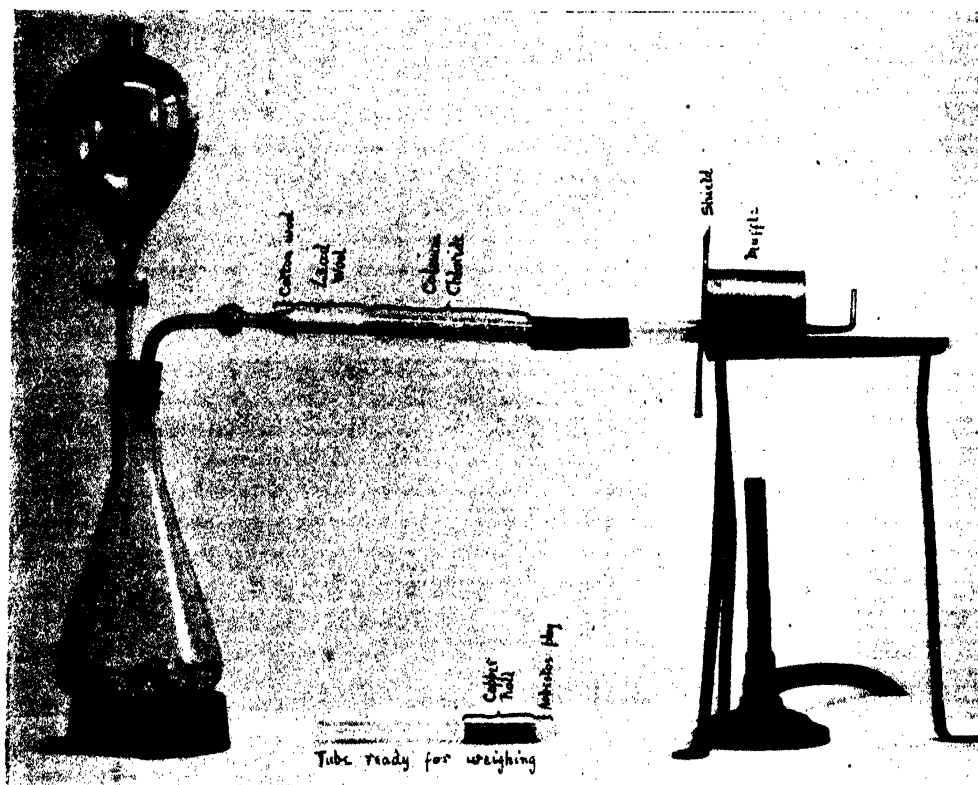
8 EVANS: A NEW PROCESS FOR THE ESTIMATION OF ARSENIC, WITH
 A NEW PROCESS FOR THE ESTIMATION OF ARSENIC, WITH NOTES ON
 THE CHEMISTRY OF THE MARSH-BERZELIUS PROCESS.

By B. S. EVANS, M.C., B.Sc., F.I.C.

(Read at the Meeting, December 8, 1919.)

THE process about to be described was worked out to provide a rapid gravimetric method of estimating arsenic in organic compounds.

General Principles.—The arsenic is evolved as AsH_3 , which is passed over ignited copper, the gain in weight of the copper being a measure of the amount of arsenic present.



DETAILS: (a) *Apparatus.*—The apparatus required is practically that used in the Marsh-Berzelius test. A flask, containing zinc coated with cadmium, as in Chapman's modification, closed with a double-bored rubber stopper carrying a tapped funnel and a tube containing wool soaked in lead acetate and dried and granulated calcium chloride; to this drying tube is attached, by means of a piece of vulcanised rubber tubing, a tube similar to that used in the Marsh-Berzelius test, but the drawn out portion of which is only about 2 inches long, the last inch or so being turned up.

It has been found convenient, and, in the long run, much cheaper, to have this drawn out tube made of silica, as it not only retains its shape when heated, but also can be used repeatedly (the work carried out for this paper was all done with one tube which was still perfectly good after considerably over one hundred estimations). Hard glass can, however, be used with quite good results.

(b) *Dimensions of Apparatus :*

Flask	...	...	...	...	capacity about 250 c.c.
Weight of zinc	...	...	...	...	40 to 50 grms. coated with cadmium by heating with a solution containing about 1 grm. of a cadmium salt.
Length of wide part of silica tube up to shoulder	...	...	...	...	about 12 cms.
Internal diameter of wide part of silica tube	...	...	...	...	0.9 cm.
Length of wide portion of drying tube	...	...	...	...	15 to 16 cms.
Internal diameter of wide portion of drying tube	...	...	...	...	1.2 cms.

These dimensions are those that happen to be convenient, but they have no special significance.

(c) *Process.*—A piece of thin sheet copper, about 4 inches by 1, and weighing about 2 grms., is heated to redness in the Bunsen flame, and allowed to cool; it is then formed into a little tube about an inch long by rolling round a glass rod. Into the silica tube is pushed a small fragment of asbestos wool (which first must be thoroughly ignited to drive off water), and it is pressed into place right up against the shoulder of the tube where it has been drawn out, which shoulder it should be just big enough to plug loosely. The copper tube is now dropped into the silica tube, and the latter is attached by means of a rubber connection to the drying tube. The silica tube is supported horizontally over a Bunsen burner, the portion where the copper is placed being covered by a little sheet iron muffle about $1\frac{1}{2}$ inches long, and a shield, made of asbestos card with a slot cut in it, is hung on to the silica tube, where the muffle ends, to protect the rubber connection from the heat of the burner.

Dilute sulphuric acid is now run in through the tapped funnel, and when the hydrogen evolved has displaced all the air in the apparatus, the jet and the burner are lighted, the burner being so placed as to heat that portion of the tube where the copper tube is to bright redness. The apparatus is allowed to run for two or three minutes, the burner is then turned out, and the tube is allowed to cool. When thoroughly cool, the silica tube is disconnected from the apparatus, the hydrogen replaced by air by connecting it for a moment to a filter pump, and it is weighed. After weighing, it is reconnected to the apparatus, the hydrogen started again with a little dilute sulphuric acid, and the burner and jet relighted. When the tube is again red hot the solution containing the arsenic to be estimated is run in through the tapped funnel, care being taken not to introduce any air, and the funnel is rinsed with water.

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The arsenic coming off in the form of arseniuretted hydrogen is completely retained by the copper, forming a lustrous silver white arsenide. Even if the hydrogen is evolved fast enough to cause a jet flame of an inch or more in height, not a trace of arsenic gets through, the colour of the flame affording a sensitive indicator, as the slightest trace of arsenic produces the lilac discoloration. After the test solution has all been added, the apparatus is kept running for forty-five minutes by the addition of small quantities of strong sulphuric acid (owing to the pressure in the apparatus it is best to force this in by blowing through a tube passing through a cork in the mouth of the tapped funnel). The height of the jet flame should be kept as near $\frac{1}{2}$ inch as possible. At the end of forty-five minutes the burner is turned out, and when the tube is quite cold it is disconnected, and, the hydrogen having been displaced by air as before, it is again weighed. The difference between the two weights divided by 0.986 gives the amount of arsenic added to the apparatus. The amount of arsenic present for the test should be 0.05-0.50 grm. calculated as As_4O_6 .

There is no need to clean out the silica tube between tests : all that is needed is to drop out the copper and replace by a fresh roll. The same asbestos plug serves for many tests, and is, in fact, better after being used once or twice, owing to a slight danger of its not having been completely dehydrated by the first ignition. Iron and many heavy metals interfere with the estimation, and, if present, they must be separated, preferably by distillation of the arsenic.

To test the method, several estimations were carried out on different amounts of pure arsenious oxide, with the following results :

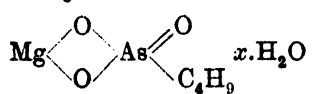
As_4O_6 taken. Grm.	As_4O_6 found. Grm.	Percentage of Arsenic recovered.
0.0750	0.0740	98.66
0.0750	0.0740	98.66
0.2030	0.1996	98.33
0.2274	0.2243	98.60
0.2634	0.2592	98.42
0.3078	0.3044	98.91
0.5030	0.4966	98.72

As a check on the purity of the arsenious oxide used, the arsenic in it was estimated both by the magnesium arsenate method and by the iodine titration method, with the following results :

$Mg_2As_2O_7$ method	...	Mean of four analyses, 100.17 per cent.
Titration		99.59 ..

From these results it would seem that the sample was practically pure arsenious oxide, and consequently the factor 1/0.986 was adopted to correct the results of the method.

The following table shows the results obtained by this method in the analysis of a variety of arsenic compounds. The organic compounds were in all cases oxidised by Thomson's method, published in the *British Food Journal*, October, 1902; the sample of arsenic trichloride was treated in the same way, and it may be noted that no loss of arsenic resulted. The second column gives the percentage of arsenic calculated direct from the gain in weight of the tube; the third column this number divided by 0.986; the last column the theoretical percentage:

Compound.	Arsenic found, per Cent.	Arsenic corrected by 0.986 Factor, per Cent.	Arsenic calculated, per Cent.
As(C ₆ H ₅) ₂ Br	{ 23.87 23.86	{ 24.21 24.20	24.27
AsC ₆ H ₅ Br ₂	23.46	23.79	24.04
C ₃ H ₇ AsBr ₂ (crude)	25.59	25.95	26.99
„ (purified)	26.45	26.83	26.99
„ (another purified sample)	26.72	27.09	26.99
C ₄ H ₉ AsBr ₂ (crude)	24.72	25.07	25.68
„ (purified)	25.17	25.52	25.68
C ₄ H ₉ AsCl ₂	36.36	36.87	36.95
(C ₆ H ₅) ₂ AsCl	27.77	28.16	28.35
C ₆ H ₅ AsO	43.83	44.45	44.65
Pure elemental arsenic	98.45	99.84	100.00
AsCl ₃ *	{ 41.11 41.03	{ 41.69 41.61	41.33
 x.H ₂ O	—	26.35	26.28 calculated from Mg.

In conducting the estimation of arsenic by the Mg₂As₂O₇ method, some facts concerning that process came to light.

Since the textbooks differed considerably among themselves as to the amount of the solubility factor to be allowed for the filtrate and washings, it was decided actually to estimate the arsenic left in solution, by the method described in this paper; the procedure adopted was as follows: The arsenate was precipitated in accordance with the directions of the textbooks, and washed with dilute ammonia, the filtrate and washings being measured and preserved. The precipitate was then dissolved in hydrochloric acid, and reprecipitated with ammonia, filtered through a Gooch crucible, washed as before, ignited to Mg₂As₂O₇, and weighed, the filtrate and washings, as before, being measured and kept. The two filtrates were evaporated separately (except in one instance, where they were mixed), acidified, reduced with SO₂, and run off by the method described in this paper.

* The sample was contaminated with a trace of As₄O₆. One per cent. of As₄O₆ would account for the excess As found.

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The results obtained are shown in the following table :

Weight of As_4O_6 taken.	Weight of $\text{Mg}_2\text{As}_2\text{O}_7$ obtained.	First Filtrate. Vol. Arsenic.		Second Filtrate. Vol. Arsenic.		Percentage of Arsenic—		
						From Pre- cipitate.	From Filtrate.	Total.
0.2964	0.4548	133 c.c.	0.0006	185 c.c.	0.0044	98.02	2.23	100.25
0.2072	0.3142	369 c.c.	0.0050	Filtrates mixed		96.85	3.19	100.02
0.1554	0.2292	175 c.c.	0.0014	225 c.c.	0.0055	94.21	5.86	100.07
0.3136	0.4672	217 c.c.	0.0024	248 c.c.	0.0098	95.17	5.13	100.30

Two points are sufficiently clear from these results : (a) The second filtrate, containing no magnesia mixture, dissolves almost four times as much arsenic as the first, although the volume is very little greater. (b) The amount of arsenic remaining in solution is not by any means so negligible as some textbooks infer. By applying certain solution factors it is possible to arrive at a correct result, yet it must be largely due to chance, unless the volumes of both filtrates and both sets of washings are carefully watched.

The missing 1.4 per cent. in the copper-tube method still remains rather a mystery, although a good deal of work has been done to trace it. A variety of possibilities suggest themselves, and were examined : (a) The missing amount of arsenic might be precipitated in the flask, either as element or as an arsenide. (b) A proportion of arsine might remain in solution. (c) The arsenic might go into solution as a non-reactive complex. (d) A proportion of arsine might diffuse through the rubber connections. (e) A proportion of arsine might be decomposed in passing through the lead wool and calcium chloride. (f) A proportion of arsine might escape from the apparatus altogether, not being held by the copper. (g) The loss might be due to steady loss of weight by the silica tube and the copper.

A, b, and c were tested in several experiments, of which the following is typical : 0.5152 gm. of As_4O_6 was run off and 97.59 per cent. was recovered (0.3809 gm. of As). A black precipitate of elemental arsenic separated on the top of the liquid in the flask (this, as will be shown later, is apt to happen when too much arsenic is used). The test was run until all the zinc was dissolved. The liquid was filtered, the precipitate treated with strong nitric acid, which completely dissolved it ; 5 c.c. strong sulphuric acid were added, and the solution was evaporated till the sulphuric acid fumed ; it was then diluted with water, reduced with sulphur dioxide, and run off ; an amount of arsenic corresponding to 1.38 per cent. was recovered (0.0054 gm. As). The original filtrate containing the zinc sulphate was evaporated down with fuming nitric acid and strong sulphuric acid and digested for several days, taken up with water, reduced, sulphuretted hydrogen passed through all night, the precipitate filtered off, boiled with nitric acid and 5 c.c. strong sulphuric acid to fuming-point of the sulphuric acid, taken up with water, reduced, and run off.

The amount of arsenic recovered was 0.0002 gm., corresponding to 0.05 per cent. Results : 97.59 per cent. + 1.38 + 0.05 = 99.02, total percentage of As recovered.

This and other similar experiments, using different methods of attack, but always with the same results, seemed to show that if the arsenic remained in the flask at all it was as a singularly obstinate complex.

To test *d*, the question whether arsine could diffuse through the rubber connection, the ordinary rubber connection in which the silica tube was pushed right up to the drying tube, was replaced by a length of about a foot of rubber tubing, on the assumption that far more diffusion should take place under these circumstances. Amount of As_4O_6 taken, 0.2952 grm.; weight of As recovered, 0.2202 grm.; corresponding to 98.47 per cent.

To test *e*—i.e., whether the lead wool or CaCl_2 caused decomposition of the AsH_3 —the drying tube was replaced by one 5 feet long, packed with lead wool and CaCl_2 in the same proportions, on the assumption that far greater decomposition should take place with greater length. Amount of As_4O_6 taken, 0.1974 grm.; weight of As recovered, 0.1480 grm.; corresponding to 98.97 per cent.

Another test was made with an ordinary drying tube in which the lead wool was replaced by ordinary cotton-wool. Amount of As_4O_6 taken, 0.2410 grm.; weight of As recovered, 0.1804 grm.; corresponding to 98.81 per cent.

With regard to *f*, the non-coloration of the hydrogen flame, and its failure to deposit arsenic on cold porcelain, were taken as proof that no arsenic was lost by passing out of the apparatus.

With regard to *g*, the various blank tests invariably showed agreement, within the errors of weighing, between the weights of the tube before and after running.

The compound formed with the copper usually melts and has a solder-like appearance, but, occasionally, brilliant silver-white blade-shaped crystals are formed.

Some analyses were attempted:

	Cu.	As.	Total.
Crystalline crust detached from copper ...	70.50	29.27	99.77
Button of arsenide exposed to large excess of AsH_3 ...	67.50	32.28	99.78
Calculated from increase in weight of copper when fully saturated with arsenic ...	70.23	29.77	100.00
Calculated from formula Cu_3As ...	71.78	28.22	100.00
Calculated from formula Cu_5As_2 ...	67.95	32.05	100.00

It would seem as if we were here dealing with a mixture of arsenides.

Although of course it is impossible to draw rigid conclusions as to the behaviour of the minute amounts of arsenic usually looked for by the Marsh-Berzelius test, it yet seemed worth while to investigate the chemistry of that process from the point of view of the larger amounts here involved, where quantitative results could be obtained.

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In the first place, it is interesting to note that insensitive zinc is a difficulty that occurs in this process, just as it does in the Marsh-Berzelius test, and that it can be overcome by the same means. In the early days of the process, the zinc was used without cadmium; a batch of zinc, however, came to hand, which gave low results; on coating this zinc with cadmium the difference in the results disappeared.

The following figures were obtained—one set with As_4O_6 , another with elemental As, and a third with impure $\text{As}(\text{C}_4\text{H}_9)_3$:

	As_4O_6 per Cent. of As_4O_6 .	As.	$\text{As}(\text{C}_4\text{H}_9)_3$ per Cent. of Arsenic.
Untreated zinc	92.60	93.15	27.78
h cadmium	98.60	98.45	28.88

What happened in the flask throws some light on, at any rate, one function of the cadmium. With both coated and uncoated zinc, very shortly after the addition of the arsenic to the flask, there was a brown coloration of the liquid, followed by the separation of a scum of metallic arsenic on the surface. In the case of the untreated zinc, this scum remained substantially unchanged at the end of the test, and undoubtedly represented the loss of the arsenic.

In the case of the treated zinc, the scum remained unchanged for some time; then (after about twenty minutes run), as generally happens, the cadmium sponge became detached from the zinc and floated up to the surface. As soon as the cadmium came in contact with the floating arsenic the latter began to disappear, and soon no trace of it was left. It would seem that what happens is that the arsenic and cadmium form an electric couple with the cadmium as anode and the arsenic as cathode, the cadmium dissolves, and the nascent hydrogen coming off from the arsenic combines with it to form arsine. Later work showed that the separation of elemental arsenic is a fairly common occurrence. If the amount of As_4O_6 in the flask much exceeds 0.5 grm. it seems invariably to occur, and therefore it is desirable to keep the amount of arsenic below that limit; the majority of salts, too, if present in sufficient amount, also cause a separation of arsenic; unless, however, the amount separated is excessive, the cadmium sponge, if it floats up properly, should easily deal with it. If from any cause undissolved arsenic should be left at the end of the test, it is necessary to filter it off, dissolve in strong nitric acid, add 5 c.c. of strong sulphuric acid, take down to the fuming-point of the sulphuric acid, take up with water, reduce with sulphur dioxide, and, after the latter has been boiled off, run off again, adding this result to the first one. An example is the following: Amount of As_4O_6 taken, 0.7318 grm.; percentage, calculated from first run, 94.84; percentage, calculated from second run, 3.75; total, 98.59.

A few observations were made which have a bearing on this separation of arsenic:

(a) Some powdered arsenic (element) was added to the flask, the jet being lighted but not the burner. The arsenic colour appeared in the jet flame at once,

but this colour markedly increased when the cadmium floated up and came in contact with the arsenic. (b) Some zinc was added to a solution of As_4O_6 in sodium bicarbonate; it slowly became coated with a brown film. On adding a little acid, a considerable amount of arsenic separated out. (c) Most of the zinc used in this work was sufficiently pure to cause the hydrogen to come off slowly and with difficulty. It was repeatedly noticed that addition of a solution of pure As_4O_6 caused an immediate rush of gas, just as the addition of a copper salt would.

From these observations it would appear that the first action when As_4O_6 is added to zinc and acid is a reduction to elemental arsenic on the surface of the zinc. Here it forms an electric couple with the zinc, the arsenic being the cathode. The nascent hydrogen coming off from the surface of the arsenic combines with it forming arsine. If, however, strong solutions of salts, which are known to cause the stripping of films, are present, or if the amount of arsenic is sufficient to cause a weakly adherent film, the latter is stripped and, being carried up to the surface, is removed from further action until the cadmium touches it.

In view of the disturbing effect of various substances on the sensitiveness of the Marsh test, it seemed worth while to determine the effect of varying amounts of them on the process under consideration, and, if possible, discover the cause of their interference.

Copper was the first metal selected. The method adopted was to keep the amount of arsenic approximately constant, and add varying amounts of copper sulphate; the amount of arsenic recovered was calculated as a percentage of the amount added; the amount of arsenic retained by the copper was calculated from the difference between the percentage recovered and 98.6. In every case there seemed to be a catalysis of the As_4O_6 —As reaction, and a considerable amount of arsenic separated on the surface; this, however, was always completely removed by the cadmium sponge. In addition to this, black particles separated in the liquid, remaining at the end of the test, when all the zinc and cadmium was dissolved, as a black powder at the bottom of the flask. This powder was filtered off, washed and weighed, and, in two or three cases, was approximately analysed: it appeared to be a copper arsenide of the approximate composition, Cu_3As . There was apparently no metallic copper left.

Weight of As_4O_6 .	Weight of Cu.	Weight of Arsenide and Insoluble Matter from Zn.*	Weight of As recovered.	Per Cent. of As recovered.	Weight of As lost.	Weight of As in Arsenide.
0.1996	0.0166	0.0533	0.1476	97.61	0.0015	0.0144
0.1996	0.0332	0.0670	0.1439	95.17	0.0052	
0.1998	0.0499	0.0846	0.1398	92.36	0.0094	
0.2003	0.0665	0.1036	0.1378	90.80	0.0118	
0.2002	0.1158	0.5574	0.0928	61.19	0.0567	

* This column includes any matter in the zinc insoluble in sulphuric acid, and is therefore only a rough approximation.

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It would seem, therefore, that copper prevents the formation of arsine in two ways: (a) By formation of copper arsenide; (b) by causing the separation of a large amount of elemental arsenic.

The cadmium causes the conversion of the separated arsenic into AsH_3 , unless the amount is excessive, but it is powerless to deal with the arsenide, though there is a certain amount of evidence to show that AsH_3 is given off very slowly from the arsenide.

The results obtained with iron were more difficult of interpretation. The iron was added in the form of ferrous sulphate, the other conditions being the same as in the copper experiments. The separation of arsenic in the flask was, if anything, more marked, and there was again a formation of arsenide in the form of a fine black powder. The arsenide was filtered off and weighed, but analyses of it were unsatisfactory. In all cases it was found that the great bulk of the iron remained in solution.

The following results were obtained:

Weight of As_2O_3 .	Weight of Fe.	Weight of Arsenide and Insoluble Matter in Zn.	Weight of As recovered.	Percentage of As recovered.	Weight of As retained.
0.2016	0.0193	—	0.1506	98.60	---
0.2003	0.0967	—	0.1450	95.57	0.0046
0.1996	0.1450	0.0388	0.1460	96.54	0.0031
0.1994	0.2418	0.0448	0.1325	87.72	0.0164
0.2004	0.4835	0.0746	0.1202	79.20	0.0295
0.1998	2.0144	0.1280	0.0760	50.20	0.0733

The most noteworthy thing about these results is that, although by far the greater part of the iron remains in solution, the amount of arsenic retained is approximately proportional to the amount of iron present, and the curve total iron—arsenic retained is very nearly a straight line, so also is the curve total iron—arsenide formed. This is all the more surprising in that, on attempting the analysis of the arsenide residues they were found to contain varying amounts of lead, which was at last traced to the cadmium salt used in coating the zinc; the cadmium salt had, however, been previously used for a large number of tests, and no lead arsenide or retention of arsenic had been observed before, so that the iron apparently brought about combination between the lead and the arsenic.

Weight of As_2O_3 .	Weight of Fe.	Weight of Arsenide.	Weight of As recovered.	Percentage of As recovered.	Weight of As retained.
(a) Contaminated Cd 0.2016	1.113	Not weighed	0.1278	83.70	0.0228
(b) Purified Cd 0.2010	1.113	0.0404	0.1196	78.56	0.0305

Iron alum was tried in place of ferrous sulphate, once using the contaminated cadmium salt, and once using the same salt purified from lead; the results only complicated the matter further.

It will be noticed that considerably more arsenic was retained with the pure cadmium salt than with that which contained lead. Lead was present in the arsenide from (a), and was absent from that from (b).

The effect of a few other substances on the evolution of AsH_3 was tried, the results being expressed as percentages recovered (uncorrected by the factor 0.986) of the amount of arsenic taken.

Weight.	Substance.	Weight of As_2O_3 added.	Percentage recovered.
10 grms.	Na_2HPO_4	0.1988	98.68
10 "	ZnSO_4	0.2000	98.77
10 "	Na_2SO_4	0.2012	98.42

The blank due to the substance, where necessary, was subtracted. These results show that the three substances used exerted no retaining effect.

Some work done on tin salts seemed to show that they did not exercise a retaining action, but the salts were so impure that the figures are not given.

I should like to express my thanks to Professor J. F. Thorpe and to Dr. Whiteley, of the Imperial College of Science, where this work was done, for suggestions and criticisms; and to the Chemical Warfare Department for permission to publish it.

DISCUSSION.

Dr. DYER said that it was common experience that much of the "pure" zinc now obtainable was too pure to be sensitive, even with the use of cadmium solution. This fault he had found could be corrected by alloying the zinc with about 0.2 per cent. of copper. Zinc thus alloyed was, of course, in itself, quite "insensitive." But when cadmium was used with it, it became very sensitive, and was also sufficiently brisk in its action with hydrochloric acid. Much of the trouble experienced by workers with the Marsh-Berzelius process was due to unsatisfactory calcium chloride, much of that now obtainable being strongly absorptive of arseniuretted hydrogen, especially if used for more than two experiments.



NOTE.

The Editor desires to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

DESICCATED COCO-NUT.

IN Messrs. Bolton and Revis's book "Fatty Foods," the following sentence relating to desiccated coco-nut occurs: "When examining this product the presence of sugar, starch, etc., must be sought for as adulterants."

A few months ago I analysed a sample of desiccated coco-nut, with the following results: Moisture, 3.87 per cent.; fat, 67.00 per cent.; sugar, 6.67 per cent. (by Fehling after inversion), 7.5 per cent. (by polarimeter). There was practically no copper-reducing power before inversion.

I was inclined to think that sugar had been added, as no mention is made by Messrs. Bolton and Revis of cane sugar being naturally present. Mr. C. E. Sage confirmed the amount of cane sugar present in the sample, but was of the opinion that the sugar was naturally present.

I analysed samples of desiccated coco-nut, copra, and fresh coco-nut, with the following results:

(1) Fresh coco-nut, milk, 1.5 per cent. cane sugar (by polarimeter); solid portion, after washing, 4.63 per cent. cane sugar on sample, 9.2 per cent. cane sugar on dry matter.

(2) Fresh coco-nut, solid portion: 6.16 per cent. cane sugar on sample, 13.5 per cent. on dry matter. No reducing sugar was present in either sample.

Copra (received from Dr. Dyer): Dextrose, 2.4 per cent.; cane sugar, 0.95 per cent.

Copra (received from Mr. Sage): Dextrose, 0.77 per cent.; cane sugar, 7.71 per cent.; moisture, 0.66 per cent.

Desiccated Coco-nut:

1. Purchased: Dextrose, traces only; cane sugar, 4.48 per cent. (4.5 per cent. on dry matter).

2. Received from Mr. Sage: Moisture, 3.3 per cent.; dextrose, nil; cane sugar, 4.56 per cent. (5.2 per cent. on dry matter).

From these figures it is evident that desiccated coco-nut may contain as much as 7 per cent. cane sugar.

E. Bourquelot (ANALYST, 1904, 29, 330) gives the composition of two samples of commercial Indian sugar; one prepared from the milk of the coco-nut contained 74.95 per cent. of cane sugar.

The amount of cane sugar present in desiccated coco-nut no doubt depends on whether the solid portion has been well washed or not before desiccating.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Simple Method for Measuring the Acidity of Cereal Products: Its Application to Sulphured and Unsulphured Oats. V. Birekner. (*J. Agric. Research*, 1919, 18, 33-49.)—Of the methods proposed by other workers, that of Schindler has been much used in America (*Zeitsch. Landw. Versuch.*, 1909, 12, 721-756). The material is extracted for sixteen to eighteen hours at normal temperature with neutral alcohol (sp. gr. 0.86), and the filtrate titrated with standard alkali. The author finds the chief deficiencies of this method to be the following: (1) Lack of a definite end-point in titration; (2) the values obtained with raw unsulphured cereals are always too high; (3) the values for sulphured cereals (oats) are too low; (4) the increase in acidity caused by sulphuring often appears as an actual decrease owing to incomplete extraction of the acid; (5) expense due to use of pure alcohol. In place of this prolonged alcohol extraction it is proposed to employ ice water for one hour (oats), or one and a half hours for maize. About 15 grms. of the sample are used for each estimation, the whole being well shaken every fifteen minutes, and kept at a temperature of 1 to 2° C., both during the extraction and while filtering. Phenolphthalein is used as an indicator, and results are expressed as the "acidity value," which is the number of c.c. $\frac{N}{1}$ alkali required to neutralise the extract from 1,000 grms. of material; for the actual titration 25 c.c. of the filtrate is titrated with $\frac{N}{20}$ sodium hydroxide.

Unsulphured oats by this method show acidity values ranging from about thirteen to twenty, while sulphured oats rise to about thirty or more. It also appears that the amount of acid present in oat kernels does not change markedly during the early stages of spoilage. Sulphur-bleached oats showed no increase in acidity upon prolonged standing in the ground state when tested by the ice-water method, since the acid-forming ferments present in the grain are destroyed by the sulphur fumes. With Schindler's method pronounced increases in the acid values were observed in such cases owing to the continued formation of certain protein cleavage products, which in aqueous solution are amphoteric, but in alcohol solution are acid. Ice water extracts of oats or maize if filtered and kept at 1 to 2° C. for twenty-four hours without being neutralised undergo no change in acidity, but, if neutralised, a new formation of acid takes place notwithstanding the low temperature. H. F. E. H.

Crismer Value of Butter Fat. L. Vandam. (*Ann. Falsific.*, 1919, 12, 260-269.)—The alcohol used in determining the Crismer value (critical temperature of solution in alcohol) of butter fat should have a sp. gr. of 0.7976 at 15° C., corresponding to 99.1 per cent. of alcohol by weight, and the strength of the alcohol should be tested from time to time by means of a standard mixture of petroleum and vaseline. The critical temperature of solution of this mixture in 99.1 per cent. alcohol is determined once for all, and, by its use, the strength of fresh quantities of

alcohol may be ascertained. If an alcohol containing more or less than 0.9 per cent. of water is employed, the results obtained must be corrected in order to give the correct Crismer value. To the value obtained is added the following correction for each 0.1 per cent. of water below or above 0.9 per cent.: From 0 to 0.4 per cent., +1.0; 0.5 to 0.8 per cent., +0.9; 1.0 to 1.1 per cent., -0.9; 1.2 to 2.3 per cent., -0.8; 2.4 to 2.9 per cent., -0.7. If the butter fat under examination is not neutral, a correction for the acidity must be added to the Crismer value; this correction is the number of c.c. of $\frac{N}{10}$ sodium hydroxide solution required to neutralise 4 grms. of the butter fat.

W. P. S.

Estimation of Added Water in Milk. J. Goldan. (*Ann. Chim. anal.*, 1919, 1, 342-345.)—The "simplified molecular concentration constant," when corrected for the volume of casein and fat as described by Ferris (*ANALYST*, 1918, 43, 35) is a trustworthy constant for the detection of added water in milk. The author has not met with a genuine milk having a value below 70, the average value being 74.

W. P. S.

Estimation of Gelatin, contained, together with Coagulable Proteins, in Feeding-Stuffs, etc. H. Wagner and G. Schöler. (*Landw. Versuchs-Stat.*, 1918, 92, 171-180; through *J. Soc. Chem. Ind.*, 1919, 38, 841A.)—Five grms. of the substance are boiled with 200 c.c. of water for five hours in a 300 c.c. Erlenmeyer flask, a larger flask being employed only with materials which "bump." The whole is then transferred to a 500 c.c. measuring flask, cooled, made up to volume, and filtered. If absence of dissolved protein in the filtrate is shown by failure of the reaction with acetic acid and potassium ferrocyanide, and of the xanthoprotein reaction, the nitrogen in 100 c.c. of the filtrate, corresponding with 1 gm. of the substance, is determined by the Kjeldahl method: this represents gelatin- plus amide-nitrogen. Another quantity of 100 c.c. of the filtrate is treated in a 250 c.c. measuring flask with 40 c.c. of 10 per cent. tannin solution (the nitrogen-content of which is determined by a blank experiment), the liquid being made up to volume, mixed, allowed to settle overnight, and filtered. The nitrogen in 200 c.c. (0.8 gm. of substance) or 150 c.c. (0.6 gm.) of the filtrate is determined by the Kjeldahl method: the result, corrected for the nitrogen of the tannin solution, gives the amide-nitrogen, and subtraction of this from the gelatin- plus amide-nitrogen yields the gelatin-nitrogen alone. The method has been tested with mixtures of blood-albumin and gelatin, and is found to give satisfactory results. A number of different feeding-stuffs have been analysed by the method.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Toxicity of Cacao Shell. Marchadier and Goujon. (*Ann. Falsific.*, 1919, 12, 283-287.)—Several French Army horses died after being fed with cacao shell and cacao shell powder in place of oats. The animals received 1.5 kilos. of the substance per day for four days before their death. Since cacao shell may contain up to 0.9 per cent. of total alkaloids (caffeine and theobromine), the horses may have taken

daily some 13 grms. of alkaloid, or over 50 grms. in all. The animals undoubtedly died as the result of alkaloidal poisoning. It is considered that cacao shell is a dangerous fodder; if the alkaloids are extracted previously, the cacao shell loses its food value owing to the simultaneous removal of other extractives. W. P. S.

Detection of Hydrocyanic Acid in Cases of Poisoning. L. Chelle. (*Comptes rend.*, 1919, **169**, 852-854.)—It has long been known that hydrocyanic acid cannot be detected in the organs of the body a short time after death. Experiments described in detail show that this is due not to the disappearance of the hydrocyanic acid, but to its fixation by the sulphur compounds produced during cadaveric putrefaction and its conversion into thiocyanic acid. This acid remains in the residue when the finely divided material is distilled with water and phosphoric acid to separate hydrocyanic acid, and the latter may be quantitatively recovered by treating the residue with a suitable oxidizing agent such as sulpho-chromic acid. Further experiments have shown that thiocyanic acid is not formed in the ordinary process of putrefaction. C. A. M.

ORGANIC ANALYSIS.

New Method for Estimating the "Copper Value" of Cellulose and the Valuation of the Quality of Cellulose. E. Hägglund. (*Papierfabr.*, 1919, **17**, 301-305; through *J. Soc. Chem. Ind.*, 1919, **38**, 895A.)—Schwalbe's method for the estimation of the copper value may be simplified by using Bertrand's volumetric method for the titration of the reduced cuprous oxide. Four grms. of the finely disintegrated cellulose are stirred with 200 c.c. of water, which is then heated to boiling, and 100 c.c. of boiling Fehling's solution added. The mixture is boiled for fifteen minutes, and then filtered in a "Neubauer platinum crucible," or else on a double filter paper with the assistance of kieselguhr paste. The cellulose containing the cuprous oxide is washed with hot water, and then treated with 100 c.c. of boiling ferric sulphate solution containing 50 grms. of ferric sulphate and 200 grms. of sulphuric acid per litre. The iron solution should previously be tested for inertness towards permanganate. The cuprous oxide is thereby dissolved, and the cellulose is washed several times with boiling water. The filtrate is then titrated with $\frac{N}{10}$ permanganate, and shows a sharp end-point from green to pink. With regard to tests for the quality of unbleached Mitscherlich sulphite pulp, the author has determined the copper sulphate absorption values, but finds no connection between these and the bleaching capacity of the pulps. The bleaching quality, however, shows a more consistent relationship with the lignin value as determined by Klason's method. None of the usual chemical tests, such as copper value, hydrolysis value, etc., showed any satisfactory concordance with the strength and mechanical qualities of the cellulose. Only in certain very pronounced cases can any definite relation be found between the tensile strength and the degree of digestion of the pulp. It may frequently happen that a fully digested pulp is as strong as or stronger than an under-digested material.

Estimation of Cyanogen Compounds in Concentrated Ammonia Liquor. Part II. The Estimation of Thiocarbonate. P. E. Spielmann and H. Wood. (*J. Soc. Chem. Ind.*, 1919, **38**, 369-370t).—A method described previously by the authors (*ANALYST*, 1919, **44**, 143) for the estimation of cyanogen compounds in ammonia liquor has been extended to include the estimation of thiocarbonate which is usually present in the liquor. The method now proposed depends on the fact that digestion for forty-five minutes at 70°-75° C. will convert thiocarbonate completely into thiocyanate without affecting the cyanide, and that ammonium polysulphide at 30°-35° C. will convert cyanide into thiocyanate in thirty minutes without attacking the thiocarbonate. The estimations are carried out as follows, 25 c.c. of the ammonia liquor being used in each case: *Thiocyanate*.—As described previously (*loc. cit.*). The solution should be cooled with ice during the addition of the sulphuric acid. *Cyanide and thiocyanate*.—The liquid is digested at 30° C. for thirty minutes with an excess (2 c.c.) of ammonium polysulphide. *Thiocarbonate and thiocyanate*.—The liquid is diluted with 15 c.c. of water, 10 c.c. of concentrated ammonium polysulphide are added, and the mixture is heated at 75° C. for forty-five minutes. *Ferrocyanide*.—This is estimated from the amount of "blue" obtained in the estimation of the thiocyanate and of the thiocarbonate and thiocyanate. If, on acidifying the solution after digestion, precipitated sulphur obscures the red coloration formed by the addition of iron alum, an excess of the latter should be added, the mixture heated at 40° C., and filtered. Large quantities of hydrogen sulphide and slimy precipitated sulphur, formed after the polysulphide treatment, may be removed by a preliminary shaking with lead carbonate. In the above estimations the flasks containing the reacting substances should be provided with loose stoppers in order to exclude atmospheric oxygen. Some experiments were made on the removal of thiocarbonate from ammonia liquor, although this is not required in the above methods. It was found that if the liquor was shaken with lead carbonate, filtered, and the filtrate shaken with zinc carbonate, the whole of the thiocarbonate could be removed without affecting the thiocyanate, which remained in solution as a zinc salt. On boiling the filtrate, ammonia was expelled and zinc oxide separated out and was removed by filtration. The preliminary treatment with lead carbonate avoided the production of an excessive amount of zinc sulphide.

W. P. S.

Electrometric Method for the Estimation of Ferrocyanides. G. L. Kelley and R. T. Bohn. (*J. Amer. Chem. Soc.*, 1919, **41**, 1776-1782).—Ferrocyanides may be estimated by determining the change in the oxidation potential during titration with permanganate, with the aid of apparatus similar to that previously described, and with the addition of a voltmeter (*ANALYST*, 1916, **42**, 108; 1917, **43**, 373). About 1 gm. of potassium ferrocyanide is dissolved in 250 c.c. of water, the solution acidified with 2.5 to 5.0 c.c. of sulphuric acid (sp. gr. 1.58), and titrated with $\frac{N}{20}$ permanganate solution. The permanganate solution is added slowly, with constant mechanical stirring, and finally, as the end-point is approached, drop by drop, and time is allowed for the completion of the reaction. The end-point is taken as the greatest change in potential corresponding to the addition of equal

amounts of permanganate solution. If the end-point has been passed, the solution may be titrated back with standard ferrocyanide solution. Ferricyanides, or chlorides, up to 1 gm. of sodium chloride or its equivalent, do not interfere with the estimation, but salts which form precipitates with either ferrocyanide or ferricyanide must not be present.
C. A. M.

Physical Properties of Mannite and its Aqueous Solutions. J. M. Braham. (*J. Amer. Chem. Soc.*, 1919, **41**, 1707-1718.)—It is recommended that the following values, based on experimental determinations and comparison with the most trustworthy recorded results, should be adopted as physical constants of mannite in place of those given in the textbooks: Melting-point, $166.05^{\circ}\text{C}.$; specific rotation, $[\alpha]_{\text{D}}^{25^{\circ}\text{C.}} = -0.244 \pm 0.002$ and $[\alpha]_{\text{D}}^{25^{\circ}\text{C.}} = -0.209 \pm 0.002$ (calculated); sp. gr., 1.487 (room temperature); specific heat, $C^{(28-100^{\circ})} = 0.327$, and $C^{(14-26^{\circ})} = 0.315$; and heat of combustion, 4.00 cal./gram. The solubility in water (100 c.c.) was found to be as follows: At $0^{\circ}\text{C}.$, 10.36; at $20^{\circ}\text{C}.$, 18.6; at $50^{\circ}\text{C}.$, 47.6; at $80^{\circ}\text{C}.$, 115.0; and at $100^{\circ}\text{C}.$, 197.0 grms.
C. A. M.

Modification of the Phenylhydrazine Method of Determining Pentosans. P. Menaul and C. T. Dowell. (*J. Ind. and Eng. Chem.*, 1919, **11**, 1024-1025.)—A method for the estimation of furfural may be based on the precipitation of the furfural with phenylhydrazine and the estimation of the excess of phenylhydrazine in the filtrate by oxidation. Experiments in this direction showed that a quantitative evolution of nitrogen could be obtained by the action of ammoniacal copper sulphate, provided chlorine or other halogen ion were absent. The distillation was therefore carried out with sulphuric acid instead of hydrochloric acid, using sodium sulphate instead of sodium chloride to lower the solubility of the precipitate. During the distillation the volume in the flask was kept practically constant by the addition of water. Phenylhydrazine was added to an aliquot portion of the distillate, agitation for the necessary time being effected by bubbling carbon dioxide through it. A portion of the filtrate from the hydrazone was introduced by means of a dropping funnel into a 250-c.c. Fresenius nitrogen bulb which had previously been filled with ammoniacal copper sulphate and heated to expel the air. A 100-c.c. burette was connected to the tube of the Fresenius bulb, and a glass tube provided with a stop-cock connected the neck of the bulb with a Schiff's nitrometer containing sulphuric acid. The measured portion of the filtrate was brought into the bulb through the dropping funnel by closing the cock leading to the nitrometer and lowering the burette. The bulb was then heated so as to keep the reaction mixture near the boiling-point until the reaction was complete. The results by this method coincided exactly with those by the phloroglucinol method, and the time required for an estimation of the excess of phenylhydrazine in the filtrate is only about twenty minutes.
J. F. B.

Method for the Direct Estimation of Naphthalene in Tar, Tar Oil, and Crude Naphthalene by Conversion into its Picrate. Knoblauch. (*J. Gasbel.*, 1918, **61**, 136-137, 145-149; through *Chem. Zeit. Rep.*, 1919, **43**, 247.)—The alcoholic

solution of the substance is treated at 60° C. with an excess of solid picric acid, and the heating continued until all is in solution. The mixture is then cooled slowly, diluted with 0.3 per cent. picric acid solution to 120 c.c., the precipitated picrate collected on a filter, and washed with 0.2 per cent. picric acid solution. The filter is folded over the precipitate, pressed slightly, placed in a beaker, 80 c.c. of water are added, and the picric acid titrated with $\frac{N}{10}$ sodium hydroxide solution, using methyl-orange as indicator. A small correction must be made for the amount of picric acid wash-water retained by the filter-paper.

W. P. S.

Estimation of Unsaponifiable Substances in Crude Materials for Soap Manufacture. Herbig. (*Seifenfabr.*, 1919, **39**, 174; through *Chem. Zeit. Rep.*, 1919, **43**, 239.)—Goldschmid's method of estimating unsaponifiable substances in soap material (*Seifenfabr.*, 1918, **38**, 41-42) may be modified by making the first extraction by shaking with 100 c.c., the second with 50 c.c., and the third and fourth extractions with 20 to 30 c.c. of ether. The colour of the ethereal extract does not necessarily indicate that unsaponifiable substances are present, since in test experiments the percentage results agreed, although the last extract was still coloured. As a rule, two extractions (by shaking) with ether are sufficient, but in the case of certain kinds of fats three or four extractions are necessary.

C. A. M.

Estimation of Volatile Fatty Acids. G. Wiegner and J. Magasanik. (*Chem. Zeit.*, 1919, **43**, 656.)—The authors discuss the question of the distillation of volatile fatty acids and the connection between the concentration of a fatty acid-water mixture and its vapour pressure, and state that a vapour-pressure curve for a propionic acid-water mixture, constructed by Konowaloff, does not agree with results obtained by Duclaux and by the authors themselves. For a distillation interval in which the fatty acid and water vapour pressures alter linearly, the following equation applies: $\log y = k \log x + 2 - 2k$, where y is the quantity of acid in the distillation residue expressed as a percentage of the original amount of acid, x the quantity of water in the residue expressed as a percentage of the original amount, and k a constant. A new graph is suggested in place of the Duclaux curve, the abscissæ being the difference of the logarithms of the quantities of water in the distillation residue, and the ordinates the difference of the logarithms of the quantities of acid in the residue; k may be found from this graph. In homologous series and for dilute solutions, k increases by 0.6 for each additional CH_2 group in the molecule. The constant, therefore, increases as the fatty acids become less soluble, or, in other words, the least soluble acids are the more volatile in steam. If 2 per cent. solutions of the fatty acids are distilled until the volume is reduced by one-half, the following quantities of the different acids, respectively, will be found in the distillate: Formic acid, 22.7 per cent.; acetic acid, 36.6 per cent.; propionic acid, 58.5 per cent.; *n*-butyric acid, 73.8 per cent.; *n*-valeric acid, 82.4 per cent.; *n*-caproic acid, 88.2 per cent.; *n*-caprylic acid, 94.8 per cent.; *n*-capric acid, 97.7 per cent. For the distillation of acetic acid at 25° to 30° C. under 14 mm. pressure, $k = 0.287$. Steam distillation of fatty acids, when the volume of water, w , is kept constant, follows the

equation $\log y + \frac{kx}{w} = 2$, where y is the residual percentage of acid, x the grms. of water vapour, and k the constant.

W. P. S.

INORGANIC ANALYSIS.

Electrometric Analysis with Potassium Ferrocyanide. E. Müller. (*Chem. Zeit.*, 1919, **43**, 597.)—Electrometric titration with potassium ferrocyanide solution fails in the case of many metals owing to the fact that the precipitated ferrocyanide is not of definite composition. Lead and zinc may, however, be titrated accurately by this method; there is a sharp change in potential for the compounds $\text{Pb}_2\text{Fe}(\text{CN})_6$ and $\text{K}_2\text{Zn}_3[\text{Fe}(\text{CN})_6]_2$. In the case of zinc, the end-point is more sharply denoted by the change in potential than by the use of uranium nitrate as an external indicator.

W. P. S.

Method for the Analysis of Nitrous Gases. A. Geake and F. J. Squire. (*J. Soc. Chem. Ind.*, 1919, **38**, 367-369r.)—The following method is described for the analysis of gases in a system of absorption towers used in connection with a plant for the denitration of nitroglycerin spent acid. The gases consisted of a mixture of nitric oxide and peroxide, carbon monoxide and dioxide, oxygen, and nitrogen. The sampling device consisted of a glass bottle of known capacity, and of such size that the sample of gas contained about 0.3 gm. of nitrogen oxides. The bottle was closed with a stopper, through which passed a tapped glass tube, the diameter of the bore of the tap and of the outer portion of the tube being 2 to 3 mm. The portion of the tube inside the bottle was bent so that it nearly touched the wall of the bottle near the shoulder. Twenty-five c.c. of $\frac{N}{2}$ permanganate solution and 1 c.c. of concentrated sulphuric acid were placed in the bottle, the air exhausted from the latter, and the tap then closed. To take the sample of gases, the tube was inserted in the sampling hole and the bottle tilted, so that the permanganate solution covered the end of the tube inside the bottle. The tap was then opened for one second (but not long enough to completely fill the bottle with the gases) and then closed. The volume of the residual gases in the bottle at atmospheric pressure was determined by connecting the tapped tube with an Orsat apparatus, the burette and levelling tube of which were filled with mercury, then making the measurement in the usual way. The portion of the gases drawn into the Orsat apparatus was used for the estimation of carbon dioxide (absorption in sodium hydroxide) and oxygen (by phosphorus). The residual gas in the apparatus was taken as being nitrogen; actually it contained carbon monoxide, but as this is inactive and has the same density as nitrogen its presence was unimportant. Air was then admitted to the sample bottle, the permanganate solution titrated with ferrous sulphate solution, and the total combined nitrogen estimated as described by Bowman and Scott (*ANALYST*, 1915, **40**, 474).

W. P. S.

Colorimetric Determination of Lead Dioxide in Litharge. W. V. Morgan. (*J. Ind. and Eng. Chem.*, 1919, **11**, 1055.)—Lead dioxide in litharge

may be determined approximately by noting the intensity of the colour produced by the oxidation of aniline. The method is also applicable, with slight alterations, for the determination of oxidising agents in general where they occur as an impurity in non-oxidising agents. A definite amount of litharge—*e.g.*, 5 grms., is boiled for a minute with a solution containing 2 grms. of aniline hydrochloride in 10 c.c. of water and 5 c.c. of concentrated hydrochloric acid. The solution is then cooled to separate any lead chloride which has formed, and the deposit is filtered off. It is not necessary to convert the whole of the litharge into lead chloride, as the action depends on the oxidising power of the dioxide only. The colour of the filtrate is compared with that produced by known quantities of lead dioxide. J. F. B.

Separation of Iron, Aluminium, Chromium, Glucinum, Titanium and Zirconium by Means of Sodium Carbonate. P. Wenger and J. Wuhrmann. (*Ann. Chim. anal.*, 1919, 1, 337-339.)—The method is the same in principle as that described previously by Wenger and Wunder (*ANALYST*, 1912, 37, 373) for the separation of aluminium from glucinum (beryllium). The ignited and weighed oxides are mixed in a platinum crucible with six times their weight of sodium carbonate, and the mixture is kept in a state of fusion for two hours. After cooling, the mass is boiled with water, the insoluble portion collected on a filter, ignited, and again fused with sodium carbonate and extracted with boiling water. The aqueous solution (filtrate) now contains the aluminium and chromium in the form of sodium aluminate and sodium chromate, respectively. The aluminium is precipitated as hydroxide by treating the boiling solution with an excess of ammonium nitrate, added in small portions at a time, and is separated by filtration. The chromate in the filtrate from the aluminium hydroxide is reduced by alcohol and precipitated as hydroxide by ammonia. The portion remaining insoluble after fusion with sodium carbonate contains the iron, titanium, zirconium, and glucinum as oxides. If titanium is present it is impossible to separate the iron and zirconium oxides by treatment with hydrochloric acid (1 : 1), since the presence of this metal causes a part of the zirconium oxide to pass into solution; this method of separation, however, may be applied in the case of iron and zirconium oxides alone. Further, iron and titanium oxides may be separated by any of the usual methods, even in the presence of glucinum, provided that zirconium oxide is not also present. Iron and glucinum oxides may be separated by fusion with potassium bisulphate and subsequent treatment with potassium hydroxide solution. W. P. S.

Method for Estimating Phosphorus in Steel containing Vanadium. J. Maitchell. (*Chem. News*, 1919, 119, 212.)—Two grms. of the steel are weighed into a 500 c.c. flask, and, when dissolved in 90 c.c. of nitric acid (sp. gr. 1.2), $\frac{N}{10}$ potassium permanganate is run in until a permanent precipitate of manganese oxide is formed. The whole is then cooled to below 20° C., and $\frac{N}{10}$ ferrous ammonium sulphate is added until the solution is bright green, and all the brown precipitate is dissolved. Twenty c.c. of ammonia (sp. gr. 0.96) are then added, and the solution shaken until the precipitated ferric hydroxide is all redissolved,

care being taken to keep the whole below 20° C. Sixty c.c. of faintly ammoniacal ammonium molybdate solution are next added, and, after closing the flask with a rubber bung, the whole is vigorously shaken for three minutes, after which the solution is allowed to stand for twenty to thirty minutes. To prevent the vanadium coming down with the phosphomolybdate and giving a dark orange precipitate, the solution must be kept below 20° C. The molybdate precipitate is next filtered off, washed free from acid, shaken up in 80 c.c. of water and estimated by running in excess of standard caustic soda, and titrating back with sulphuric acid and phenolphthalein. The whole determination can be done in three-quarters of an hour, unless the carbides are difficult to get into solution. Analyses are adduced, showing the great accuracy of the method as applied to test mixtures. H. F. E. H.

Deroode Method for Determining Potash. T. E. Keith and H. E. Shiver. (*J. Ind. and Eng. Chem.*, 1919, 11, 1049-1052.)—In a previous paper the authors had shown (*ANALYST*, 1918, 43, 236) that the Lindo-Gladding method gives low results for water-soluble potash in certain cases, and that the Deroode method afforded a simpler and more accurate procedure without the use of platinum apparatus. The validity of the method has now been further investigated in relation to the possible presence of sodium nitrate, ammonium salts, and high percentages of phosphates and organic matter. The Deroode method (*ANALYST*, 1898, 23, 278) as finally applied is carried out as follows: Ten grms. of material are transferred to a 500 c.c. flask, 300 c.c. of water added, boiled for thirty minutes, cooled, made up to the mark, and filtered through a dry filter, the first runnings being discarded. An aliquot portion of 50 c.c. is placed in a thin porcelain dish of 200 to 250 c.c. capacity, and 30 c.c. of *aqua regia* are added after putting the dish on the hot plate. After evaporating to dryness another 30 c.c. of *aqua regia* are added and again evaporated to dryness; the residue is taken up with a small volume of hot water, 10 c.c. of concentrated hydrochloric acid are added and the solution is again evaporated to dryness. The residue is taken up with hot water and platinic chloride solution is added. When the solution is almost evaporated to dryness the dish is preferably taken off the water bath and twirled round so as to cover the dried portion with the liquid portion and so avoid a baked condition. The precipitate is covered with acidulated alcohol as previously described (*loc. cit.*) and allowed to stand for about an hour, the precipitate being thoroughly broken up. The precipitate is collected on a tared filter, washed with acidulated alcohol until the runnings are colourless, and then at least seven times with 10 c.c. portions of Lindo's ammonium chloride wash. The washing is continued five times with 80 per cent. alcohol, washing to the top of the funnels. The method has been tested in the presence of ammonium salts greatly in excess of the quantity likely to exist in commercial samples. It has also been tested in the presence of sodium nitrate, phosphatic matter and organic matter (cottonseed meal), and the results recorded are entirely satisfactory. J. F. B.

Method for Bringing Elementary Sulphur into Solution for Analysis. A. P. Bjerregaard. (*J. Ind. and Eng. Chem.*, 1919, 11, 1055.)—Most samples of sulphur leave a considerable residue insoluble in carbon bisulphide, and boiling with

nitric acid and bromine leads to the formation of fused globules difficult to attack, while bromine water causes the evolution of sulphurous gas. The procedure recommended by the author takes advantage of the solubility of sulphur in dry liquid bromine, the solution then being oxidised by concentrated nitric acid. About 0.1 grm. of sulphur is dissolved in 1 c.c. of bromine, and 10 c.c. of nitric acid are added. After removing the excess of bromine and nitrous fumes by heat, 100 c.c. of water and a few c.c. of hydrochloric acid are added and the solution is again boiled to expel the nitric acid. The sulphuric acid formed is then precipitated in the hot solution by barium chloride in the usual manner.

J. F. B.

Estimation of Zirconium by the Phosphate Method. G. E. F. Lundell and H. B. Knowles. (*J. Amer. Chem. Soc.*, 1919, **41**, 1801-1808.)—An experimental study of the conditions necessary for the quantitative precipitation of zirconium as phosphate has shown that the separation is complete from cold or tepid solutions containing 2 to 20 per cent. by weight of sulphuric acid, provided that an excess of from 10 to 100 per cent. of the theoretical amount of diammonium phosphate be used for the precipitation. The hydrolysis which takes place when the zirconium phosphate is washed with water may be prevented by washing the precipitate with a cold 5 per cent. solution of ammonium nitrate. Ignition of the precipitate washed in this way yields zirconium pyrophosphate, the zirconium oxide (ZrO_2) factor for which is 0.4632, but the compound obtained by igniting the precipitate after washing with water is not constant in composition. Zirconium can be quantitatively separated from iron, aluminium, chromium, cerium, and thorium, by precipitation as phosphate from its solution in 20 per cent. (by weight) sulphuric acid, whilst a satisfactory separation from titanium can also be effected by adding sufficient hydrogen peroxide to keep the titanium in solution.

C. A. M.

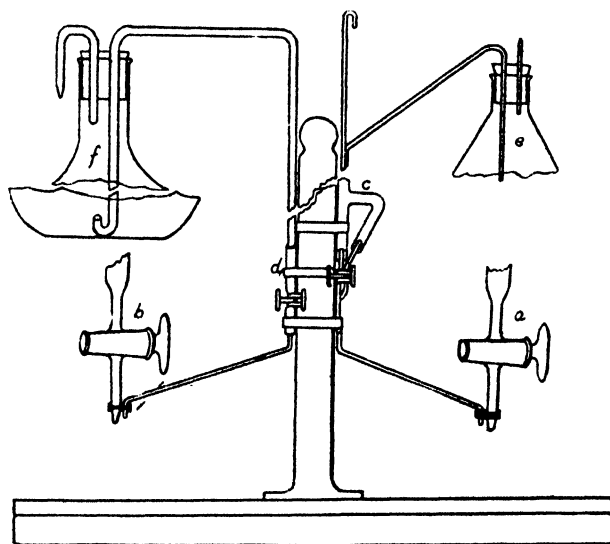
APPARATUS, ETC.

Determination of Ignition Temperatures by the Soap-Bubble Method. A. G. White and T. W. Price. (*J. Chem. Soc.*, 1919, **115** and **116**, 1248-1264.)—The soap-bubble method for the determination of ignition temperatures was first described by McDavid (*J. Chem. Soc.*, 1917, **111**, 1003), who obtained results in which the experimental error is assumed to be less than $\pm 3^\circ \text{C}$. His results are very high in comparison with other recent determinations. The authors have applied the method to determine the ignition temperatures of mixtures of ether, benzene, light petroleum, and hydrogen with air, and describe in great detail the conditions under which consistent results can be obtained. Even after careful standardisation of the coils used, size of bubble, and numerous other details, the results were found to be effected by the physical state of the igniting surface and the nature of the material of which it is made, alteration in the results obtained being observable even upon the addition of small quantities of the salts used for standardisation purposes, thus discrediting the method as one capable of giving the true ignition temperature of a gas mixture. The ignition temperature of a particular gas mixture

as determined by two different coils often varied by more than 150°C. , and results indicated the unreliability of the method even for comparative purposes.

H. F. E. H.

Apparatus for Rapid Gastric Analysis together with a Method for the Preservation of Starch Solution. R. J. Miller. (*J. Ind. and Eng. Chem.*, 1919, 11, 963-964.)—In gastric analysis the following estimations are required at frequent intervals: Total acidity—titration with $\frac{N}{100}$ potassium hydroxide. Free acidity (Sahli method)—titration with $\frac{N}{100}$ sodium thiosulphate, finishing with starch solution. Formol titration or amino-acid—titration with $\frac{N}{100}$ potassium hydroxide after the addition of neutral formalin. The arrangement of apparatus illustrated in the figure has been devised as a permanent equipment for making these tests. The installation consists of two standard burettes of 30 c.c. graduated to 0.05 c.c., the burette, *a*, on the right being connected with the reservoir of standard alkali, and that on the left, *b*, with the thiosulphate. The burettes are fitted with a device for

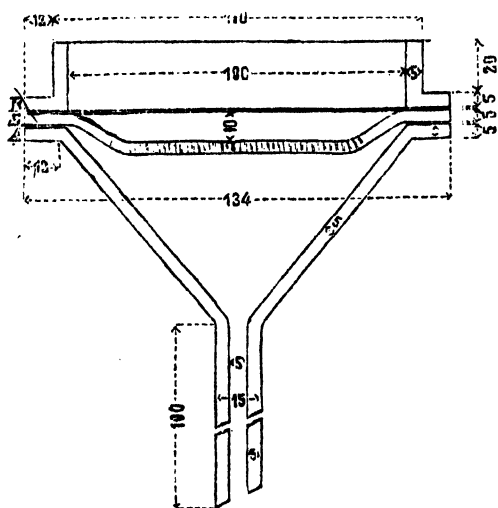


automatic filling to the zero mark when suction is applied to a branch tube. Attached to the upright portion of the burette-stand, by means of adhesive tape, is an automatic pipette, *c*, connected with the formalin reservoir, *e*, the pipette being controlled by a two-way pinchcock arranged so that as soon as the charge of formalin is delivered through the discharge-pipe leading to the point of the burette, *a*, the pipette automatically fills up again from the reservoir to its predetermined level and is ready for the next discharge. The attachment shown at *d* is also bound to the upright by tape, and is an ordinary siphon controlled by a pinchcock and connected with the reservoir, *f*, for starch solution, which is delivered as required to the point of the thiosulphate burette, *b*. For the preparation of a stable solution of soluble starch, 10 grms. of soluble starch are ground to a paste with cold water and added

to 750 c.c. of water which has been boiling for fifteen minutes. The mixture is heated for ten minutes and diluted with boiling water to 1 litre. The storage reservoir is rinsed with boiling water and the starch solution added, being immediately covered with a layer of $\frac{1}{4}$ inch of liquid paraffin. If desired, the hot starch may previously be shaken with 1 c.c. of toluene. The siphon is charged while the solution is still hot, and the reservoir is preserved in a sterile condition.

J. F. B.

Chemical Analysis with Membrane Filters. R. Zsigmondy and G. Jander. (*Zeits. anal. Chem.* 1919, 58, 241-280.)—As a development of the collodion film ultra-filter, the authors have introduced a parchment-like membrane which has the appearance of glazed paper or white glacé kid, and which is prepared by the

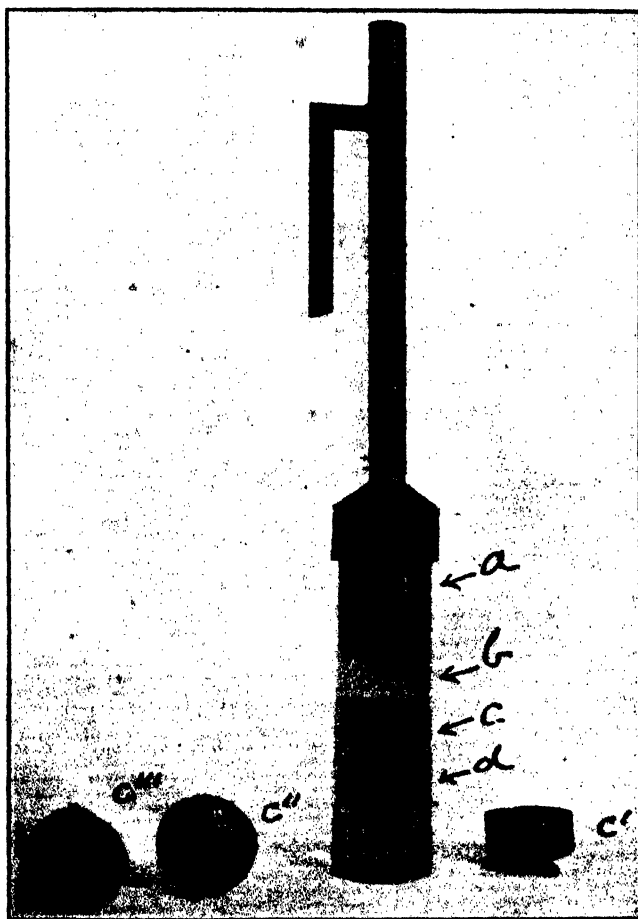


evaporation, under special conditions, of somewhat complex solutions of certain colloids. These filters, under the action of the suction pump, are permeable to water with a wide range of rapidity, but possess the property of retaining the finest and most gelatinous precipitates, and will even filter out colloidal suspensions of graphite, the filtered residue being readily and completely removable from the glazed surface. The membrane filters are used for all kinds of filtration in quantitative work in conjunction with an apparatus (see Fig.) resembling the Buchner funnel with a dished filter-plate. The funnel is made in three pieces which are held together with

screw clamps and with rubber rings between the parts. A smaller funnel has been designed to take the place of the Gooch crucible, and has a filter-plate of watch-glass shape representing a segment of a sphere. The filtering membranes are classified according to the rate of passage of 100 c.c. of water through a circular area of 100 mm. diameter, and the most useful kind for quantitative work ranges from twenty to fifty seconds. In using the filter the liquid is decanted at a uniform rate into the middle of the hollow, but must never come within 0.5 cm. of the joint. The precipitate is then brought on to the filter and drained by suction, and it is to be noted that, whereas the precipitate must be well drained, it must not be sucked so dry that cracks appear in the cake. These filters differ from paper filters in that none of the finer particles penetrate into the pores, but all remain free on the glazed surface. Filtration is facilitated if a paper filter be fitted against the plate immediately beneath the membrane. It is not desirable either to dry the membrane filter in the oven or to incinerate it; the best procedure is to remove the precipitate quantitatively into a crucible. This may be done either immediately in the wet condition or after drying in a suitable manner. Precipitates which dry to a loose powder are best handled moist. For this purpose the screw clamps are

loosened and the membrane is removed, bent round in the form of a scoop, and the main portion of the precipitate is pushed into the crucible with a horn spatula. The filter is then spread out over a deep crystallising dish, and the residue of precipitate washed down with a few drops of water and a small paint-brush into the hollow thus formed. The fluid is tipped into the crucible, and the last traces of precipitate are removed from the spatula and brush in a similar manner. The water is evaporated from the crucible on a water bath. In the case of gelatinous precipitates, the whole filter should be dried in the oven at 80° C. until the precipitate is partially dry but still semi-coherent. In this case the membrane must be kept under tension during drying, substituting rings of paper for the rubber-packing rings. J. F. B.

Fat Extraction Apparatus. J. M. Pickel. (*J. Ind. and Eng. Chem.*, 1919, 11, 1053-1055).—The author has devised a form of extraction apparatus with which

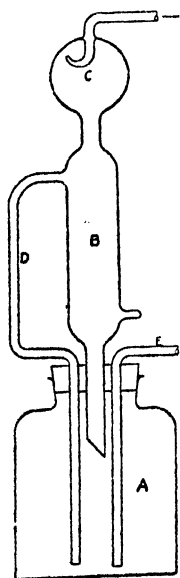


twenty analyses may be conducted simultaneously on an electric hot plate, 4½ ins. × 24 ins. About 15 c.c. of ether are used, of which one-third to one-half is

recovered. The change from extraction to recovery is effected by merely giving the condenser a slight turn on its axis. This condenser is made of metal, with cold water inlet extending down the centre, and the outlet passing up around it to an overflow discharging into a trough. The extraction is carried out in a cylindrical glass jar, *b*, $5\frac{1}{2}$ ins. high and $1\frac{9}{16}$ ins. in diameter inside. This is just sufficient to take the body of the condenser, which measures $1\frac{1}{2}$ ins. outside at the part marked *a*. The lower portion of the condenser, on which the condensation takes place, is narrowed down to $1\frac{7}{8}$, and the lower surface is tapered to an eccentric apex, or drip point, from which the condensed ether falls into the cup, *c*. This cup contains a funnel leading right through it, so that when the condenser is so arranged that the ether drips into the funnel, it passes through into the crucible beneath, whilst, if the condenser is turned so that the drip misses the funnel, the ether is collected in the cup and is recovered when the cup is removed. The cup and funnel are made of metal. The substance to be extracted is contained in the crucible, *d*, of alundum, and the drip of ether is distributed by placing a sheet of paper or perforated metal over the substance. The crucible is supported by lugs extending from the side of the glass cylinder, and the fat extract falls through the crucible to the bottom of the cylinder. If desired, a receiver of aluminium may be fitted in the bottom of the glass cylinder to receive the ethereal extract, in which case the lugs are omitted, and the crucible is supported in the upper part of the receiver.

J. F. B.

Nitrogen Generator for Laboratory Use. W. L. Badger. (*J. Ind. and Eng. Chem.*, 1919, 11, 1052-1053.)—The following description relates to a simplified

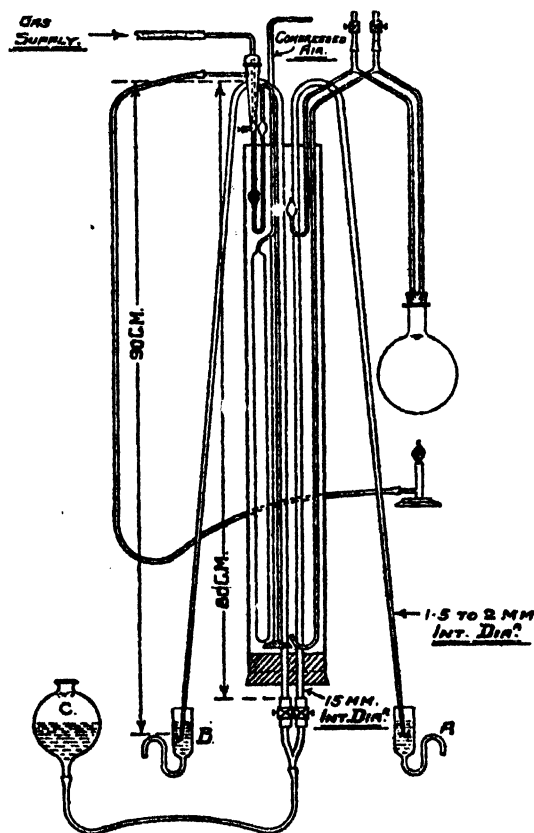


form of Van Brunt's apparatus for preparing nitrogen free from oxygen by the action of copper in the presence of an ammoniacal solution of an ammonium salt. A wide mouth bottle, *A* (see Fig.), is filled with the reagent and as much metallic copper as can be got into it, preferably in the form of straight vertical wires. *B* is a Liebig condenser shell with the lower water connection sealed up; it is filled with copper turnings or punchings. A tube, *D*, is connected to the upper water connection, and extends nearly to the bottom of the bottle, *A*. At the upper end of the condenser should be a bulb, *C*, although the trap as shown is not absolutely necessary. The gas inlet tube, *E*, should also reach nearly to the bottom of the bottle. A fourth tube may be added for blowing out spent reagent if desired. The bottle should hold 2 litres of reagent. This reagent is made by diluting 1 part of commercial ammonia with 1 part of water, and saturating the mixture with ammonium chloride. This reagent remains active until such a heavy precipitate is formed that the apparatus is clogged. A slow circulation of liquid takes place through the tube, *D*, and prevents premature clogging of the tower. The tower is preferably charged with 5 inches of copper punchings, which present a much larger

surface than turnings. Air or 95 per cent. commercial nitrogen is passed through the apparatus in a steady stream, and the ammonia carried over is absorbed in Drechsel washing bottles.

J. F. B.

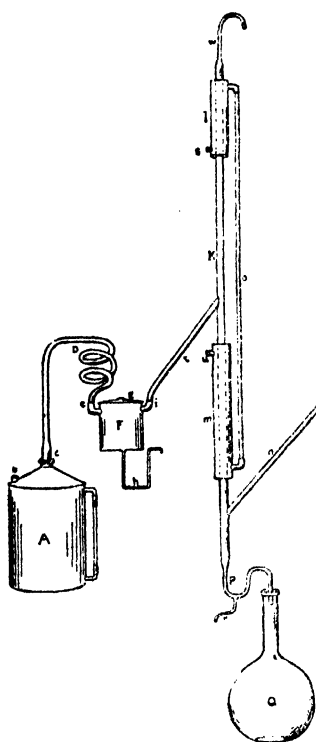
Apparatus for the Estimation of Vapour Pressures. A. Morton. (*J. Soc. Chem. Ind.*, 1919, 38, 363-364r.)—The apparatus shown in the diagram is constructed without stopcocks, thus obviating errors caused by organic solvents dissolving the lubricants used for the taps. Two capillary tubes are fused to the top of the barometer tubes, and bent downwards so that their ends dip into the mercury reservoirs A and B. After the mercury in the apparatus has been dried with strong sulphuric acid the excess of the latter is forced from the barometer tubes into A and



B by raising the reservoir C, and removed from A and B by means of a pipette and filter-paper. This treatment with acid is repeated until the mercury is at the same level in both tubes, and the distance between this level and that of the mercury in the reservoirs is equal to the barometric level at the time of the experiment. The liquid under examination is introduced into A, the screw clip on the left-hand tube closed, A lowered until the end of the capillary is immersed only in the liquid, and a sufficient quantity of the latter drawn up into the barometer tube by first raising and then lowering C, after which A is raised again so that the end of the capillary tube is again immersed in the mercury. The reservoir C is then lowered below the level of A, so as to keep the liquid in the barometer tube under reduced pressure, and the apparatus is kept at a constant temperature by means of a water-

jacket provided with a thermo-regulator and constantly stirred by means of compressed air. Bubbles of air escaping from the liquid are forced back into A by raising C and escape through the mercury seal. The reservoir C is again lowered, the left-hand clip opened, and the difference between the levels in the two tubes is measured with a cathetometer. After lowering C still further, the difference in the levels is again measured, and if the readings do not agree the vapour and air in the liquid under examination are forced back into A again, and this treatment is repeated until concordant results are obtained at different levels. C. A. M.

Method for the Preparation of Conductivity Water. C. B. Clevenger. (*J. Ind. and Eng. Chem.* 1919, 11, 964-966.)—The apparatus illustrated in the figure is a modification of the Bourdillon still, in which the steam is purified by means of



a current of pure air. The boiler, A, of about 15 litres capacity, is connected with the block-tin spiral D and the trap F, in which the spray and less volatile impurities are collected and discharged through h. The $\frac{3}{4}$ -inch tube, c, extends to within $\frac{1}{2}$ inch from the bottom of the trap, and the steam emerges from i, passing through the pipe, l, into the upright condenser, K, which is a block-tin pipe $1\frac{1}{4}$ inches in diameter, and 6 feet long. Copper cooling jackets, l and m, are connected together by the glass tube, o. The container, q, of glass or block-tin, is attached to the syphon, P, by a rubber stopper; a syphon or water-tap is provided for drawing off the water, and a guard-tube of soda lime to purify the air which enters. The outlet, r, closed by a tin stopper, is used for drawing small samples for testing. The main air stream enters through a purifying system attached to n, and is preferably produced by means of a small rotary pump. The air is purified by passing through two large bottles containing sulphuric acid and broken glass, and two similar bottles containing concentrated sodium hydroxide solution; afterwards it passes through a vertical column of glass beads, and, finally, through an empty bottle serving as a trap. The tube, w, at the top of the condenser, is also provided with a train of guard bottles, to prevent

access of carbon dioxide and ammonia. In operation, the water in the still is first boiled for a short time to expel gases, and then a few c.c. of phosphoric acid are added. About one-fourth of the steam condenses in the trap and carries away impurities. The steam entering K is purified by the upward air-stream, and is cooled just to condensation by the upper jacket, l. As the hot water runs down the sides of K, it continually meets air, which is less and less contaminated, and is finally cooled by the lower jacket, m. The speed of the air current is only limited by the efficiency of the purification apparatus, and the air escaping at w carries away the volatile

impurities of the water. All parts which are in contact with steam are either of block-tin or tin-lined ; permanent connections are soldered, but movable connections are luted with tin-foil. The output of the still is about 1 litre per hour of water, having a conductivity value of 0.8×10^{-6} reciprocal ohms.

J. F. B.



REVIEWS.

CHEMICAL REAGENTS : THEIR USES, METHODS OF TESTING FOR PURITY AND COMMERCIAL VARIETIES. By DR. C. KRAUCH, Chemist to the Firm of E. Merck. Darmstadt. Second English Edition, Revised and Enlarged by H. B. STOCKS, F.I.C. Pp. vi + 368. London : Scott, Greenwood and Son, 1919. Price 17s. 6d. net.

A good selection of reagents is considered in this book. Of course, no one will find it covers their every need, but those excluded are reagents of secondary importance. Thus among omissions are *o*-tolidine, silver lactate, sodium citrate, aluminium chloride, mannitol, cadmium sulphate, and Sudan III.

While dealing with the detection of, and proposing limits for, impurities, the work is very methodical and quite good. There is some cold-blooded restraint, which is probably intentional ; for a liturgy on the catastrophes that will arise from, say, ammonia in sulphuric acid, lead and iron in zinc, carbonate in ammonium hydroxide, and oxidisable matter in acetic acid, would be quite easy to begin, but it would be without end. On this, the main purpose of the book, comments of a trivial order only are called for. As a limit of acidity in 40 per cent. formaldehyde, 1 c.c. is required to show no acid reaction after adding one drop of normal alkali. This is liberal, because the reviewer has never met a batch requiring half as much alkali. Not one in ten samples of the cream of tartar met with in this country will, after the addition of ammonia, give no colour with hydrogen sulphide.

Under paragraph headings of "Commercial Varieties," the names and composition of chemicals of inferior quality are given, which adds to the usefulness of the book.

It is evident that only the laboratory worker is considered, as the uses of a substance in the arts and technical processes are seldom mentioned. The general plan for "Uses" is this : The reader is told that a substance is used for one purpose, and referred to a published paper ; and for another purpose, and referred to a text-book. The full scope of a reagent is often not fully recognised ; and even where references or descriptions of tests are given, they are not indexed. Thus, at least two tests for furfural are mentioned, one of which is also described. Neither is indexed, and, to find them, one must search till they are found in monographs devoted to sulphuric acid and xyldine respectively.

Glycerol is regarded solely as a boric acid reagent, alkanet only as an indicator, picric acid is confined to alkaloid, albumin and naphthalene reactions, and the uses

of benzidine in food analysis and enzyme work are omitted. The last is regarded solely as a sulphuric acid and a blood reagent. It is probably not often used now for the latter purpose, except for occult blood in fæces.

Starch is the only substance with which a simple iodine solution is said to give a colour. Formaldehyde is regarded solely as an antiseptic; without agreeing entirely with some optimists among American physiological chemists that distillation in Kjeldahl's process can be avoided by the use of formaldehyde (the reviewer's experience is that results on the low side are usually obtained), there are nevertheless several conditions when the attraction of amino groups and ammonia for it may be utilised. There are the "aldehyde figure" for proteins, Sorensen's method for amino-acids, E. W. Brown's method for ammonia in urine, and it has also been recommended by Krapivini for the estimation of ammonia in effluents and sewage. The suitability of iron alum for salicylic acid is not mentioned, and ferrous sulphate is only a nitrate-test reagent.

Actual errors are few and include a statement that the stain produced on mercuric chloride paper by arseniuretted hydrogen is unaffected by light or moisture, and the necessity of drying turmeric paper to bring out the red due to boric acid is overlooked. It is not meticulous accuracy to say all salts of barium are poisonous, as five ounces of the sulphate are given as a radiographic meal.

Methyl violet is selected for the detection of mineral acid in vinegar, and tro-pæolin OO paper for hydrochloric acid in gastric juice, but there is no direction to warm the test-paper in the latter case to reveal small amounts. There is no mention of dimethyl-amino-azo-benzene, which is the most popular indicator for estimating hydrochloric acid in gastric contents. In a recognition of the work of Lubs and Clark, phenol-red is mentioned. This is the indicator which has quite recently been adopted by the Medical Research Committee, National Health Insurance, for the hydrogen ion concentration of bacteriological media.

The references to "Commercial Organic Analysis" are to editions published during Mr. Allen's lifetime; there is a reference to the second edition of Clowes and Coleman's "Quantitative Analysis"; and there are only five references to papers in all the last twenty volumes of the ANALYST.

A Tralles alcohol table is provided. Tralles got his order for this article from the Prussian Government in 1811, since when a gravity lower than 0.7946 at 60° F. has been found and accepted for 100 per cent. alcohol. A reader desiring an interpolated table is referred to Hehner's tables in Muspratt's "Handbuch der technischen Chemie." These tables were published in Vol. V. of the ANALYST, and subsequently reprinted in pamphlet form by J. and A. Churchill. There is no reference to Thorpe's compilation of 1916.

If the reviewer has read correctly between the lines of the Preface, no foreign assistance has been invoked since the publication of the former edition in 1902.

WILLIAM PARTRIDGE.

THE CHEMIST'S YEAR-BOOK, 1918-19. Edited by F. W. ATACK. Fourth Edition.

Two vols., pp. 1146. Sherratt and Hughes, Manchester. Price 15s. net.

The fourth edition of this admirable reference book has made its appearance (previous editions havin been reviewed in this Journal: see ANALYST, 1915, 40, 427; 1917, 42, 406), and the editor and contributors are to be congratulated upon their success in the production of such an invaluable *vade-mecum*, which continues to receive further useful additions with each succeeding edition. The new sections added to the present volumes include "Analysis of Clays, Firebricks, and Silica Materials," by J. W. Mellor; "Agricultural Chemistry," by E. J. Russell; and "Crystallography," by E. H. Rodd; while several of the older sections have been revised, and the tables giving the properties of inorganic and organic substances have been entirely reset, with a decided increase in utility.

The annual revision to which this work is subjected still leaves some matters of minor importance outstanding. Considering the value in many industries of the estimation of arsenic in various materials, it is to be expected that one at least of the principal methods employed should be given in some detail, but the reviewer has failed to discover any reference whatever throughout the volumes. Further omissions, which might readily be rectified, occur in the table "General Properties of Organic Compounds," from which the densities of various common substances, such as trichlorethylene, vanillin, phenolphthalein, etc., are missing.

A few of the tables, although correctly indexed, are misplaced; for example, the "Solubilities of Bromine" on p. 540 appear in the section devoted to the solubilities of gases in liquids, while the "Solubility of Ether" occurs on p. 555 under the heading "Solubilities of Solids in Liquids." These two tables, together with the "Solubility of Aniline" on p. 973, might be included in a small section by themselves. The Temperature Conversion table—Centigrade to Fahrenheit—on p. 420 should immediately follow the corresponding table on p. 418. In the table "General Properties of Inorganic Substances," pp. 142-208, the editor has followed the usual unsatisfactory plan of expressing solubility by the terms "soluble," "slightly soluble," etc. The futility of this method may readily be demonstrated by requesting several chemists to express their numerical conceptions of these terms, when the results will be found extremely variable. Some endeavour should be made to give approximate values, or else definite limits should be given to the above terms. The contraction "diss.," which occurs occasionally in the last column of the above tables, will present no difficulty to the trained chemist; but for the benefit of the many other users of this book, the definition of this contraction should be provided at the bottom of p. 142.

Some of the tables will not meet with the use their importance deserves, for the reason that no references to them are given in the index. Among these are the pressure conversion table on p. 408, the "Delicacy of Certain (Alkaloid) Precipitating Reagents," p. 1049, and the "Boiling-Points of Glycerine Solutions" on p. 613. The latter is certainly included under the index title of "Glycerine Solutions: Vapour Pressure of," but no indication is given that the boiling-points are also furnished. There is no apparent reason why the two tables giving the boiling-points of alcohol solutions on pp. 619 and 939 should be indexed, the one under the

letter "A" and the other under "E" (Ethyl Alcohol). Both should obviously appear under the heading of Alcohol.'

The book is almost free from serious errors, but the following should be rectified: On p. 383 the table "Measures of Capacity" (B.P., 1914) gives a centilitre (Cl.) as equivalent to 0.01 c.c. (0.169 minims), and 1 decilitre (Dl.) equal to 0.1 c.c. (1.69 minims). In earlier editions of the book these equivalents are given correctly, but in slightly different form, and the error has evidently crept in during transcription. The table on p. 441, giving the specific gravity of acetic acid solutions, still retains the inaccurate figure of 1.0007 for the sp. gr. of a 1 per cent. solution, but for this error the author of the table and not the editor is to be held responsible. However, in spite of the above faults, the book is of great value, and very well worth the price charged. Now that the diary of doubtful utility has been omitted, a few blank pages might be inserted at the end of Vol. II. for users to note down any corrections or suggestions which might be of service to the editor when preparing the next edition, together with a date before which such communications should be sent in.

T. J. WARD.

GAS AND FUEL ANALYSIS FOR ENGINEERS. By AUGUSTUS H. GILL. 1919. John Wiley and Sons; London: Chapman and Hall. Price 6s. net.

The author claims that his book "is an attempt to present in a concise, yet clear, form the methods of gas and fuel analysis in testing the efficiency of a boiler plant." The first chapters are devoted to apparatus and methods employed in simple flue gas analysis, and to temperature measurement—the latter a short chapter of less than three pages, which is unlikely to be of much service to anyone, so "concise" is it. Chapter V. deals with gas analysis with the Hempel apparatus. A brief description is given in the succeeding chapters to solid, liquid, and gaseous fuels. Evidently American practice with gaseous fuels is different in some respects from ours, for we learn that "coal or illuminating gas was formerly produced by the distillation of bituminous coal; it is at present made by the enrichment of water-gas."

Methods for the analysis of fuels and calorimetry follow, the Emerson type of bomb calorimeter (a form practically unknown in this country) being selected. The method described of taking half the loss by radiation per minute as the basis for the cooling correction is unsatisfactory for a calorimeter capable of a considerable degree of accuracy. In connection with calorimetry it is surprising to find the author describing the quite discredited Berthier method (estimation of calorific value by the weight of lead reduced from litharge) and even referring to it as a "method which would seem worthy of more attention than it has received."

In discussing the calorimetry of gases it is stated that "the net heating value of a gaseous fuel represents the conditions in which by far the greater quantity of gas is consumed for cooking, heating, and gas engines, and is the one which should be reported." Possibly the author is unaware of the different and more scientific attitude adopted to-day in reference to gross and net calorific values, an attitude so well summarised by one authority as "the figure obtained by subtracting the heat due to condensation of the water was absolutely artificial and meant nothing."

The author in attempting conciseness has frequently succeeded in producing

"scrappiness." His working directions would frequently require amplification by a good teacher. The appendix contains some useful tables and specifications for coal and oil purchase.

J. S. S. BRAME.

THE ANALYSIS OF MINERALS AND ORES OF THE RARER ELEMENTS. By W. R. SCHOELLER, Ph.D., and A. R. POWELL. Pp. x+240. London: Charles Griffin and Co., Ltd. 1919. Price 16s. net.

On its title-page this book is described as for analytical chemists, metallurgists, and advanced students, and in the preface the authors say that the old arbitrary separation into common and rare elements is still adhered to by most teachers, with the result that the training of the future chemist can hardly be said adequately to prepare him for present-day requirements. It might be argued that the student would prepare himself better for his life's work, whatever it may be, by attempting to master the foundations of analytical chemistry rather than by studying methods for the separation of comparatively rare elements. If he has done that, he will have no difficulty in making use of this book or a later edition of it, if and when he is called upon to estimate one of the rarer elements. To the older professional man it would be difficult to exaggerate the value of this book. Half of us may go through our professional life without once being called upon to look for or to estimate a rare element; to a few a single group of rare elements may be as familiar as iron or carbon to the rest of us, but half of us have no such special interest, and yet may be called on to estimate rare elements as an unusual development of our ordinary work. The preliminary hunt through the literature is rendered easier than was formerly the case by the well-indexed volumes of the *ANALYST*, but is still tedious and often unprofitable unless the analyst has ready access to a good library or is the owner of an exceptional one.

It would be difficult to embark on a critical review of a book of this scope. Three-quarters of the separations described the reviewer has never attempted; many of the remainder but once or twice.* A close study of the pages he would feel confidence in criticising revealed nothing conflicting with his own experience, and will encourage him to refer to other sections of the book with confidence. No doubt some indifferent methods have been included; the authors frankly state that in such a book a certain amount of compilation is unavoidable, and that no chemist has opportunity to test all the methods covering so vast a subject. It is to be hoped that other chemists will not only refer to this book for information, but will read critically those sections which they could write themselves, and that they will find time to point out any errors to the authors; for this is a book of which a new edition will be called for every five years, supposing analytical chemistry maintains the rate of progress which marked the five years ending with 1914. And, in any new edition, one may be permitted to express the hope that for every new method included one old one may be dropped. The fact that this book is so small is one of its merits. If and when it gets to twice the size, it will probably be a worse book. It has been said that it takes ten years for a new method to get into the textbooks and twenty years for a bad one to drop out. This book contains methods described only last year, and there are signs that the authors wished to avoid the reproach of

offering a bewildering choice of alternative methods. If they maintain this attitude in future editions, and ruthlessly scrap third-best methods, the path of the next generation of analytical chemists will be smoothed.

It has been taken for granted that every chemist at all likely to be called upon to estimate unfamiliar elements—and that is half the readers of the *ANALYST*—will acquire this book, and so no detailed statement of its scope has been given. The title gives almost all the information required as to the scope of the work, but perhaps it should be added that it deals with lithium, titanium, vanadium, selenium, tellurium, molybdenum, tungsten, uranium, and the platinum metals, as well as with the still rarer elements.

G. C. JONES.

VOLUMETRIC ANALYSIS. Second Edition. By C. H. HAMPSHIRE, B.Sc. (Lond.), F.I.C. Pp. 127. 1919. London: J. and A. Churchill. Price 5s. net.

In the second edition of this little volume newer matter has been added in order to meet requirements of the British Pharmacopœia, and, although primarily written for pharmaceutical students, the contents deal with general volumetric analysis and should be useful to all young students of chemistry. The chapter on "Indicators" might with advantage be supplemented and strengthened by a summarised table, and, in dealing with examples from organic chemistry where only in some cases are molecular and constitutional formulæ given, an extension of this feature would have been more consistent.

The application of normal solutions is well expressed, and repetition of calculations is designed to compel a clear understanding of fundamental principles.

The chapters assigned to acidimetry and alkalimetry, oxidation and reduction, are exceedingly well written and free from error, and throughout the book the influence of conditions is well stressed.

More advanced students will observe omission of details underlying principles in some of the methods, notably oxidation of formaldehyde by iodine, but, having regard to the preface, serious exception cannot be taken to this, and the volume can be regarded as having fulfilled the author's claim. It is a useful little handbook.

GEO. R. THOMPSON.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE ESTIMATION OF MERCURY.

By H. B. GORDON, PH.D.

In studying the estimation of mercury in an organic mercury compound it was found that precipitation of the mercury by means of hydrogen sulphide was not applicable, as nitric acid would be used for decomposing the compound. The apparatus used in the estimation of mercury as the metal was not available. Moreover, the electrolytic precipitation of mercury in a platinum dish is liable to be injurious to the platinum, particularly if chlorides are present. It therefore seemed advisable to employ some other method than those usually described in the standard works on quantitative analysis.

A number of investigators have precipitated the mercury from solution by means of some other metal. For this purpose C. Reichard (*Pharm. Zentr.*, **51**, 443) used aluminium, and found that copper, silver, and cadmium interfered. M. Francois (*Compt. Rend.*, 1918, **166**, 950-952) precipitated the mercury with pure zinc, dissolved the zinc in sulphuric acid, and weighed the mercury. He apparently regarded this method as sufficiently accurate. The zinc used must be pure, and an estimation appears to have required about three days for its completion. Obviously, copper or silver would interfere, but these were not present in the case to which the method was applied. Various investigators have precipitated the metal by means of copper, and have then converted the metal to iodide (M. Perlstein and J. Abelin, *Münch. Wochschr.*, **62**, 1181-1183; Menière, *Bull. médical*, June 29, 1910), or have volatilised the metal on to a weighed gold plate (K. M. Vogel and O. I. Lee, *J. Am. Med. Assoc.*, **62**, 532-534).

The author thought it should be possible to estimate mercury by precipitating it upon an element higher in the electromotive series, weighing the metal and mercury, volatilising the mercury, and reweighing the metal. The loss in weight should represent the mercury, unless some of the other metal was also volatilised. For the precipitating metal, copper was chosen, because partly it is not readily volatile and partly because it is lower than hydrogen in the electromotive series, and so can be used in acid solution and will not precipitate zinc, cadmium, etc., if they should be present.

In a quantitative method the completeness of the reaction is an essential consequence. The discharging potential of mercury and that of copper from normal solutions of their ions upon the respective metals differ, as given in the literature, by about 0.42 volt. It follows, then, that if a piece of copper is placed in a solution containing mercury ions, the mercury will be displaced until the copper ion concentration exceeds that of mercury ion to such a degree that, if the ratio of these two concentrations be substituted in Nernst's formula $e = \frac{RT}{nF} \log \frac{c'}{c''}$, the value of the electromotive force, e , should be about 0.42 volt. The copper strip on which mercury has deposited should show a less tendency to displace mercury ions than should pure copper, hence a large surface of copper is advantageous to decrease this effect. In practice this effect was practically eliminated by putting a second piece of copper into the solution after the mercury had been nearly precipitated. Since the mercury is, in any case, diluted with copper when deposited on that metal, the electromotive force between the metallic mercury and the solution is not the same as if the metal were pure mercury, and the tendency for the ionic mercury to precipitate should be greater than if the metallic mercury were pure. The dilution of metallic mercury and copper by each other thus has two effects which tend to neutralise each other.

Substituting numerical values in the above equation, and bearing in mind that for the copper ion, whose discharging potential is to be decreased by increasing its concentration, the value of n , the valence, is 2, we find that at 25° C. $\frac{e'}{c''} = 1.7(10)^{14}$, or, in order that copper and mercury ions should be in equilibrium with metallic copper and mercury, the concentrations of copper ions should be $1.7(10)^{14}$ times that of the mercury ions—in other words, the precipitation is complete. To test this conclusion in practice, about 0.3 grm. of crystalline copper sulphate was dissolved in 100 c.c. of water, to which was added 0.1 mgrm. of mercuric chloride. A small piece of bright copper was placed in this solution and allowed to stand for about nineteen hours. The copper was then removed from the solution, and a dark stain, indicating mercury, was visible on its surface. It was washed with water, alcohol, and ether, and after standing for some time in a desiccator, was weighed. It was then heated in a current of dry hydrogen until the dark stain disappeared, and was cooled in hydrogen. When again weighed, it was found to have decreased in weight by about 0.1 mgrm. To be sure that the colour and loss in weight were not due to the formation of copper oxide and its subsequent reduction by hydrogen, the experiment was repeated, obtaining the same results as in the first experiment; but in the second trial the mercury was condensed in a constricted part of the tube in which the copper was heated, and, later, iodine vapour was drawn through the tube. A faint reddish tinge at the constriction of the tube showed the presence of mercury iodide. Narrow strips of bright copper were placed in other samples of copper sulphate solution in which no mercury salt had been placed. These showed no tendency to acquire a dark stain such as the copper piece showed in the presence of mercury. In another experiment about 5 grms. of crystalline copper sulphate were dissolved in water, 200 c.c. of concentrated hydrochloric acid were added, and made up with water to 2,000 c.c. To this solution was added mercuric chloride

equivalent to 0.1 grm. of mercury; a small piece of copper was suspended in the solution by a fine thread. The next day the copper showed evidence of a mercury deposit, but it was allowed to stand in all about thirty-six hours; it was then removed and treated as in the previous experiments. Again a loss in weight of 0.1 mgrm. was observed, and the dark stain disappeared during the heating. The test with iodine vapour was negative in this case, but the stain could not have been due to oxide, as it would not form in the acid solution. From these experiments it is apparent that the darkening of copper by mercury gives an extremely sensitive test for that metal if the absence of other metals that could deposit on copper is assured.

The reaction may be hastened by having a large surface of copper, a small volume of solution, and by stirring. A volume of 60 to 70 c.c. was placed in a Nessler tube, and the copper, in the form of a coil of wire gauze, was hung in the solution from a platinum wire by a hook on the end of a glass rod. This rod was rapidly raised and lowered by attaching the rod to an eccentric rotated by a small water-motor. The solution was thus stirred, and the copper brought frequently into contact with all parts of it.

Preliminary experiments showed that, if the solution is neutral, some of the mercury is precipitated as a grey powder. 2N hydrochloric acid seemed to prevent this precipitation, as did a smaller concentration of nitric acid. Nitric acid, however, even when dilute, slowly attacks the copper. 2N acetic acid did not entirely prevent this precipitation, though it would doubtless do so if the mercury solution were sufficiently dilute as indicated (see Estimation No. 4, Table 1).

In rather concentrated solutions there is a tendency for some of the mercury to be deposited in the form of a very fine powder which floats on the surface of the solution. In some of the preliminary experiments, in which this phenomenon was shown to a marked degree, this floating mercury was recovered and weighed in a Gooch crucible. In these cases it weighed about 0.1 mgrm. In the experiments recorded in the table following, the loss due to this cause was not so large—in most cases this floating mercury was not observed at all—and no effort was made to collect it.

TABLE 1.

Time No. 1.	Time No. 2.	Conditions.	Mercury found.
20 min.	50 min.	2N with HCl	4.8 mg.
20 "	140 "	2N "	4.6 "
20 "	20 "	2N "	4.8 "
30 "	30 "	$\frac{N}{2}\text{NH}_4\text{NO}_3 + \text{acetic acid}$	4.8 "
18 "	30 "	$\frac{5N}{8}\text{NH}_4\text{NO}_3 + \frac{N}{4}\text{nitric acid}$	4.6 "

In the preliminary experiments an effort was made to estimate considerable quantities of mercury—e.g., 0.2 grm.—but for this purpose rather large copper coils seemed necessary, and these were too heavy for the small water-motor to move them with a steady motion. The platinum wire available for hanging the coil from the

glass rod was of small diameter, and sometimes broke when large coils were used. Accordingly, coils weighing from 4 to 7 grms. were used, and only small amounts of mercury were estimated.

Table 1 gives the results of five consecutive estimations of mercury in a solution of mercuric chloride. A volume of 50 c.c. was used, the weight of mercury, as determined by the sulphide method, being 4.8 mgrms.

The first and second columns record the time the first and second coils were left in the solution.

The procedure was as follows: The measured volume of standard mercuric chloride solution was placed in a Nessler tube, to this was added the other solution to give the desired acidity or salt concentration, the Nessler tube was placed under the stirring apparatus, a copper coil was hung, by a platinum wire attached to it, from a hook at the end of the glass rod of the stirrer, and the rod was released, so that the coil was immersed in the solution. The water was then turned into the motor at such a rate that it gave the glass rod a fairly rapid motion. After a sufficient time had elapsed, the coil was replaced by a second one, and was washed by immersing several times in water, twice in alcohol, once in ether, and once in ether dried over metallic sodium. Upon removal from each liquid the coil was drained on filter-paper. After draining from ether, it was put in a desiccator under diminished pressure. When the second coil had remained in the solution for a sufficient time it was removed, washed in the same manner as the first coil, and also dried *in vacuo*. After sufficient time had elapsed for the ether to have evaporated from the coils they were weighed. After their weight had become constant they were placed in a hard glass tube, and a current of dry hydrogen passed through. When the air had been expelled, the coils were heated by the flame from a Bunsen burner with a wing tip, and kept at a dull red heat for about a minute, after which the flame was removed, and they were allowed to cool in a slow stream of hydrogen. They were then withdrawn from the glass tube and weighed. The loss of weight of the second coil was much less than that of the first coil. In only one case given in the above table was it as great as 0.7 mgrm., and in one case it was only 0.2 mgrm. This was to be expected, since the mercury was so completely removed by the first coil that the second coil was scarcely coloured by it. If the glass tube showed any copper on its walls as a result of heating the coils in it, this copper was estimated, and its weight subtracted from the total loss in weight of the coils due to heating. The result was the mercury found, given in the last column of the above table. In some cases no copper was seen on the glass tube. In other cases it was estimated by dissolving in nitric acid, washing into a suitable vessel, and making a colorimetric comparison with a known copper solution. In some cases the colours were matched in ammoniacal solution; in others the solution was acidified with acetic acid (after making alkaline with ammonia) and potassium ferrocyanide added, and the colours matched. In some of the later estimations, when the copper did not exceed 0.2 mgrm., it was estimated by its appearance on the tube. One tenth of a mgrm. makes a considerable stain on the glass tube. In the presence of a nitrate, especially of free nitric acid, as in determination No. 5, the coil on which the mercury was deposited had a smoother, better-looking surface than when these were absent,

and there was also less tendency for copper to deposit on the glass tube when the mercury was volatilised.

To attain constant weight in the vacuum desiccator, a coil had usually to be left in it several hours, though sometimes practically constant weight was reached in an hour. A means for drying the coils more quickly would evidently be advantageous. This was attempted by placing the coil, after draining off the ether, in an oven heated to 90° to 95° C., removing it after one minute, and cooling it in a desiccator without evacuation. In this case 4.9 mgrms. of mercury was found in 50 c.c. of the standard mercuric chloride solution. The second coil appeared to acquire a film of oxide which may account for the slightly high result. This modification of the procedure would apparently be useful when the time required for an estimation is of more importance than extreme accuracy. Under such conditions it should be possible to complete an estimation of mercury in a solution in about one and a half hours.

An attempt was made to avoid the use of the hydrogen during the volatilisation of the mercury. In this case the coils carrying the mercury were heated in the hard glass tube as usual, except that the tube was filled with air instead of hydrogen. The copper oxide formed was then reduced by placing the coil in alcohol vapour. To transfer the coil from the glass tube to the alcohol vapour without permitting it to cool, and without breaking off any of the copper oxide, requires great care. A tendency was also noted for thin flakes of copper, formed by reduction of the oxide, to shake loose from the coil. This method of reduction is therefore not to be recommended.

The usefulness of a method of analysis depends largely upon the number of conditions which interfere with its accuracy. For this reason, the effect of the presence of various substances was briefly studied. As indicated above, while the presence of nitric acid interferes with the estimation of mercury as sulphide it is rather helpful than otherwise in this method. Obviously those metals standing above copper in the electromotive series will not interfere with this method, so no attempt was made to study their effects, but such elements as arsenic, antimony, bismuth, and silver, which are displaced from solution by copper, would interfere with the estimation of mercury by this method if they were present in the solution and if they were volatilised to an appreciable extent with the mercury. To test the applicability of the method in the presence of these elements, pieces of copper gauze were placed in solutions containing the chlorides of arsenic and antimony. After some hours these were removed from the solution, were washed and dried, weighed, heated in hydrogen, and again weighed. The coil containing the arsenic lost 2.4 mgrms., and that containing antimony lost 5.3 mgrms. This was but a small part of the arsenic and antimony present. Similarly, a copper coil which had stood for some hours in a solution containing about 10 mgrms. of bismuth, in the form of chloride, lost, upon heating, about 1 mgrm. of bismuth. Two estimations were also made with conditions as in No. 5, Table 1, except that about 10 mgrms. of arsenic in the form of chloride was added to the solution. In one of these cases the mercury (and arsenic) evaporated from the coils was 7.4 mgrms. In the other case it was 5.2 mgrms. Evidently but little arsenic was volatilised in the latter case.

The effect of silver upon the results obtained by this method was also tested. A dilute solution of mercuric nitrate was standardised by this method. The conditions as to time and concentration of reagents were essentially the same as in estimation No. 5, Table 1. Three results showed 6.2, 5.9, and 6.1 mgrms. of mercury in 25 c.c. of the solution. With conditions the same as in these estimations except that about 10 mgrms. of silver nitrate were dissolved in the solution, the copper coils lost, when heated, 6.6 mgrms. As no copper was deposited on the glass tube, the vaporisation of several tenths of a mgrm. of silver was indicated.

These results indicate that the estimation of mercury by this method, in a solution containing arsenic, antimony, bismuth, or silver, would not be satisfactory. It seems not improbable, however, that by heating the copper coils in a glass tube placed in a larger glass tube, whereby the heating would be more gradual, and the temperature under better control than by the method of heating used in the present investigation, it might be possible to volatilise the mercury without driving off a weighable amount of these elements. This method of heating would probably also eliminate all loss of copper, since the copper stain was usually on a part of the glass tube which had become unduly hot.

SUMMARY.

The estimation of mercury in dilute solutions has been carried out by displacing the mercury from its compound by means of copper in the form of gauze, heating the copper in hydrogen, and noting the loss in weight due to the volatilisation of mercury.

The detection of 0.1 mgrm. of mercury in two litres of solution has been found possible by this means.

The presence of nitrates does not interfere with this method, but is rather beneficial.

The presence of arsenic, antimony, bismuth, and silver interfere with the method, but it seems probable that, with careful heating, the method might be used in their presence.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Estimation of Amino-Acids in Wine. E. Garino-Canina. (*Annali Chim. Applic.*, 1919, 12, 112-118.)—The formation of amino-acids in wine is due to the action of enzymes in yeasts or bacteria upon the proteins in the grape must or wine, or to the auto-digestion of the protoplasm of the *Saccharomycetes*. Acids of the type of leucine, tyrosine, aspartic acid, etc., may then undergo further enzymic decomposition, being converted into amyl alcohol, succinic acid, etc.; while at the same time the basic nitrogen group is liberated, and there is an increase in the amount of ammonia in the wine. Sørensen's method of estimating amino-acids by titration with formaldehyde (*ANALYST*, 1908, 33, 364) gives good results in the case of wines when used in the following manner: the wine (500 c.c.) is nearly neutralised with barium hydroxide, and colouring matters are removed by treating it with 50 c.c. of a 20 per cent. suspension of animal charcoal. It is then evaporated to half its volume and filtered, the filtrate and washings (about 400 c.c.) treated with 2 grms. of barium chloride and a slight excess of saturated alcoholic barium hydroxide solution, and the ammonia distilled, by means of a current of air, into standard sulphuric acid and titrated. The liquid is then rendered neutral to azolitmin paper, 10 c.c. of 30 to 40 per cent. formaldehyde solution (recently neutralised towards phenolphthalein) added, and the acidity titrated, as in Sørensen's method. The amount of nitrogen in the form of amino-acids ranged in twenty-six samples of wine of different origin from a trace to 91.0 m. mgrms. per litre; whilst a sample of grape juice contained 112.0 m. mgrms., and a sample of sterilised must 98.0 m. mgrms. The total nitrogen in the wines varied from 28.0 to 453.0 m. mgrms., 30 to 40 per cent. of which was in the form of proteins, about 22 per cent. as amino-acids, 2 per cent. as amides, and up to 2 per cent. as ammonia. It is pointed out that the keeping properties of wine probably depend to a large extent upon the form in which the nitrogenous constituents are present, and that, as has been shown in the case of beer, the presence of complex nitrogenous compounds may be a direct cause of turbidity.

C. A. M.

Determination of Moisture in Malt. T. J. Ward. (*J. Inst. Brew.*, 1919, 25, 335-352.)—The author describes results of work extending over a considerable period, during which time over 10,000 malt moisture determinations were made. The malt was, in all cases, ground in a coffee-mill, and dried in a jacketed oven containing boiling water. A large number of conditions upon which correct results depend are summarised, among the more important being the following: Both ventilators of the oven must be open; no leak, however small, must allow access of steam to the interior of the oven; the vessels containing the malt should be flat-bottomed, provided with well-fitting lids, and must not contain an amount of malt exceeding 0.5 cm. in height; products of combustion from the heating burner must be pre-

vented from gaining access to the interior of the oven, and all dishes containing malt must be placed on the floor of the oven but not within 5 cm. directly behind the lower ventilator; a period of at least three hours must be used for drying, and no material other than malt should be placed in the oven during this time; the door of the oven must not be opened during the drying period. Among the factors found to have no influence upon the result are the following: The internal capacity of the oven within the limits of $3\frac{1}{2}$ and 33 litres; lifting the lid of the dish between cooling and weighing; coarse or fine grinding of the malt; the relative proportions of husk and endosperm weighed into the dish; the relative humidity of the atmosphere, provided this does not exceed 95 per cent., and the period during which the malts are dried in the oven within the limits of three and eighteen hours. H. F. E. H.

Fruit of the South American Noli Palm as a Source of Oil. (*Bull. Imp. Inst.*, 1919, 17, 186.)—The Noli palm (*Elæis melanococca* Gaertn.) is similar in character to *E. guineensis*, the West African oil palm, but is smaller in size; it is common in Columbia, from which place was derived the consignment analysed. The fruits were dry, of orange-yellow or greyish colour, from 0·8 to 1 inch in length, and from 0·5 to 0·8 inch in diameter; average weight of a single fruit, 2·5 grms. The shells of the nuts were very hard, and about 0·1 inch in thickness. The kernels were similar to the West African palm kernels from *E. guineensis*, but smaller in size; average weight of a single kernel was 0·5 gm. The fruits consisted of pericarp 16 per cent., shell 62 per cent., and kernel 22 per cent.

Pericarp.—The pericarp of the sound fruits contained 8·1 per cent. of moisture and 29 per cent. of oil, equivalent to 31·5 per cent. of oil from the dry pericarp. The yield of oil from the entire fruits is therefore only 4·6 per cent. The oil itself is an orange-yellow liquid containing a fairly high proportion of stearine. It is much more liquid and paler in colour than commercial palm oil from West Africa, but is similar in taste and smell.

The comparative analyses are as follows:

	Pericarp Oil from Noli Palm Fruits.	Commercial West African Palm Oil (<i>E. guineensis</i>).
Sp. gr. at 100/15° C.	0·8636	0·8586
Solidifying-point of fatty acids	33·6° C.	36° to 46° C.
Acid value*	29·7	—
Saponification value*	199	196 to 205
Iodine value per cent.	83·5	53 to 57
Unsaponifiable matter per cent.	0·7	—
Volatile acids, soluble	0·7	0·86 to 1·87
„ „ insoluble	0·5	—

Mgms. potash per gm. of oil.

Kernels.—The kernels contained 7·2 per cent. of moisture and 45·4 per cent. of oil—a solid fat of pale cream colour of pleasant taste and smell and slightly harder than ordinary coconut oil.

Comparative analytical values were as follows :

	Noli Kernel Oil.	Palm Kernel Oil.
Sp. gr. 100/15° C.	0·8651	0·8731
Solidifying-point of fatty acids	26·9° C.	20° to 25° C.
Acid value	0·6	—
Saponification value	234	243 to 255
Iodine value per cent.	27·7	10·3 to 17·5
Unsaponifiable matter per cent.	0·8	—
Volatile acids, soluble	1·4	5 to 6·8
„ „ insoluble	3	10 to 12

H. F. E. H.

Indian Turpentine and Rosin. (*Bull. Imp. Inst.*, 1919, 17, 159-177.)—Further samples of turpentine oil and rosin obtained by the distillation of the gum-oleo-resin of *Boswellia serrata* (nat. ord. *Burseraceæ*) have been analysed and described (ANALYST, 1916, 41, 8). The average yield of gum-resin varies from 5 to 7·5 oz. per tree, and its average composition is as follows: Moisture, 10 to 11 per cent.; turpentine oil, 8 to 9 per cent.; rosin, 55 to 57 per cent.; gum, 20 to 23 per cent.; insoluble matter, 4 to 5 per cent.

1. *Turpentine Oil.*—Two samples prepared from the gum resin analysed as follows :

	1.	
Sp. gr. 15/15° C.	0·8446	0·8523
Optical rotation	+ 31° 24'	+ 25° 57'
Acid value	nil	nil
Ester value before acetylation	2·6	3·0
Ester value after acetylation	36·4	54·3
<i>Fractional Distillation :</i>		
Fraction boiling at 153° to 160° C.	89 per cent.	56 per cent.
Fraction boiling at 160° to 170° C.	} 11 " " * {	24 " "
Fraction boiling at 170° to 180° C.		20 " "

The *Boswellia* turpentine oil readily dissolves resins such as colophony, dammar, sandarac, and soft copal, and practical trials with varnishes made with the oil showed that it could be successfully employed in place of ordinary turpentine oil.

* All between 160° and 180° C.

2. *Rosin*.—Five samples of *Boswellia* rosin were examined with the following results :

	1.	2.	3.	4.	5.
Moisture, per cent.	0.7	0.9	0.7	0.8	1.1
Ash, per cent. ...	0.5	0.4	0.03	0.3	0.2
Softens at ° C. ...	72	56	68	71	65
Melts at ° C. ...	—	—	—	78	73
Acid value ...	51.5	25	55.1	52.4	50.8
Saponification value	92.0	66.0	90.2	87.8	75.6
Iodine value present	70.0	96.0	—	64.6	76.2
Sp. gr. ... {	1.082 at 20/20° C.	1.050 at 20/20° C.	—	1.077 at 18/18° C.	1.059 at 18/18° C.

All the samples were completely soluble in alcohol, ether, chloroform, benzene, and turpentine oil with the exception of No. 2, which was only partly soluble in alcohol. Varnish-making trials gave satisfactory results.

3. *Gum*.—Three samples were analysed as follows :

	1.	2.	
Ash ...	3.5	4.1	10.6
Resin (matter soluble in 95 per cent. alcohol) ...	27.6	24.0	19.3
Soluble gum (matter soluble in water) ...	50.1	56.3	44.8
Insoluble gum and dirt (exclusive of ash) ...	18.8	15.6	25.3
Matter soluble in ether (dry and free from alcohol) ...	24.4	20.1	13.2

Experiments made on the technical scale showed that the gum was much inferior to ordinary gum for use as a sizing material, and that it was unsuitable as a material for finishing textiles. It is probable that *Boswellia serrata* gum-resin, hitherto used chiefly as a substitute for *B. carteri* and *B. frereana* in the preparation of incense, has a future before it of considerable commercial importance. H. F. E. H.

Sesbania : A Feeding-Stuff from South Africa. (*Bull. Imp. Inst.*, 1919, 17, 184.)—*Sesbania* seeds (nat. ord. *Leguminosae*) are small and hard with a shiny dark greenish-brown coat. They measure about 0.1 inch in length and possess a fairly strong pea-like flavour and a slight aromatic odour. Analysis showed them to have the composition (per cent.): Moisture 9.6, crude proteins 32.9, consisting of true proteins 24.2, other nitrogenous substances 8.7, oil 6.2, carbohydrates by difference 39, fibre 10.9, ash 1.4. Hydrocyanic acid is absent. No previous experience of their feeding value in this country is available, but, provided that feeding experiments show no unsuspected disadvantage in actual use, it may be anticipated that they will be capable of replacing peas, lentils, or millet. H. F. E. H.

ORGANIC ANALYSIS.

Analytical Method for the Separation of Acetylene, Ethylene, and Benzene. W. D. Treadwell and F. A. Tauber. (*Helv. Chim. Acta*, 1919, 2, 601-607.)—A method for the successive absorption of acetylene, ethylene, and benzene vapour when these are mixed with carbon dioxide, oxygen, etc., is as follows: Carbon dioxide is removed by means of sodium hydroxide; the acetylene is then absorbed in alkaline mercuric cyanide solution (20 grms. of mercuric cyanide dissolved in 100 c.c. of $\frac{N}{2}$ sodium hydroxide solution) by shaking the gas mixture for two minutes with 5 c.c. of this solution. Ethylene is next absorbed by shaking the residual gas with 10 c.c. of acid mercuric nitrate solution (20 grms. of mercuric nitrate dissolved in 100 c.c. of $\frac{N}{2}$ nitric acid and the solution saturated with sodium nitrate, the latter preventing the absorption of benzene vapour). The benzene vapour is finally absorbed in fuming sulphuric acid in the usual way. Oxygen and carbon monoxide do not interfere with the above absorptions. Propylene is not absorbed by alkaline mercuric cyanide solution, but dissolves in acid mercuric nitrate solution and also in 25 per cent. sulphuric acid containing 10 per cent. of mercuric sulphate.

W. P. S.

Estimation of Cyanogen Compounds in Concentrated Ammonia Liquor.
II. Estimation of Thiocarbonate. P. E. Spielmann and H. Wood. (*J. Soc. Chem. Ind.*, 1919, 38, 369-370T.)—A method described previously (*ANALYST*, 1919, 44, 143) for the estimation of cyanogen compounds in ammonia liquor, based on the depth of colour after their conversion into ferric thiocyanate, has now been extended to include the estimation of thiocarbonate. The present method depends on the fact that digestion for forty-five minutes at 70° to 75° C. will convert thiocarbonate completely into thiocyanate without affecting the cyanide, and, further, that polysulphide at 30° to 35° C. will convert cyanide into thiocyanate in thirty minutes without attacking the thiocarbonate. The various estimations are made as follows, separate quantities of 25 c.c. of the liquor being used for each: *Thiocyanate as such*.—The method described previously (*loc. cit.*) is used. During the addition of the sulphuric acid the liquid should be kept cold by means of pieces of ice. *Cyanide and Thiocyanate*.—The liquid is digested at 30° C. for thirty minutes with an excess (2 c.c.) of ammonium polysulphide. *Thiocarbonate and Thiocyanate*.—The liquid is diluted with 15 c.c. of water, 10 c.c. of concentrated ammonia are added, and the mixture is digested at 75° C. for forty-five minutes. *Ferrocyanide*.—This is estimated from the amount of "blue" obtained in the estimations of the thiocarbonate and thiocyanate. On acidifying the mixtures after digestion, precipitated sulphur may obscure the red coloration of the ferric thiocyanate. In such cases an excess of iron alum is added, the mixture heated at 40° C., and filtered. If after polysulphide treatment large quantities of slimy sulphur are precipitated and much hydrogen sulphide is evolved, it is preferable to remove most of the sulphur by a preliminary treatment with lead carbonate. Provided that the estimations are carried out in conical flasks fitted with loose stoppers there is no need to exclude air from the reacting substances, the vapours and evolved gases affording sufficient protection from the atmosphere.

W. P. S.

Detection and Estimation of Cyanides and Thiocyanates in Complex Mixtures. L. Chelle. (*Comptes rend.*, 1919, 169, 973-975.)—Fifty c.c. of the solution, containing a very small quantity of cyanide, are acidified with 5 c.c. of 50 per cent. sulphuric acid, and a current of air is passed through the mixture for three hours at the rate of 25 litres per hour; the hydrocyanic acid is collected by passing the air through an absorption vessel containing 1 c.c. of potassium hydroxide solution. The cyanide in the latter solution is then detected and estimated by the ferrocyanide or ammoniacal silver iodide methods. If the test solution also contains thiocyanate, the solution remaining after the removal of the hydrocyanic acid is treated with an excess of potassium chromate. This converts the thiocyanate into hydrocyanic acid, which is removed, collected, and estimated as before. The method is applicable to the estimation of amounts of cyanide as small as 0.01 mgrm. In the presence of bromides, hydrochloric acid must be used in place of sulphuric acid; iodides, if present, must be separated previously.

W. P. S.

Identification of Dyes. E. F. Hitch and I. E. Knapp. (*J. Ind. and Eng. Chem.*, 1919, 11, 1076-1079.)—An account of the well-known methods of identifying and separating unknown dyestuffs, including colour reactions with acids, etc., dyeing tests, microscopic and spectroscopic examination, and adsorption and solubility methods.

J. F. B.

Estimation of the Strength of Dyestuffs. W. H. Watkins. (*J. Ind. and Eng. Chem.*, 1919, 11, 1079-1080.)—The author calls attention to the precautions necessary in making comparative dye tests in the examination of dyestuffs, particularly as between buyer and seller.

J. F. B.

Change of Refractive Indices of Fixed Oils with Temperature. C. H. Wright. (*J. Soc. Chem. Ind.*, 1919, 38, 392-394r.)—The change of the refractive indices of fixed oils with change of temperature varies with the change in density, and the modulus of expansion, k , may be used likewise for calculating the refractive index at any temperature, t' , from observations made at another temperature, t . The value of k is practically a constant for all the fixed oils which have been examined, and may be taken as 0.00076. The correction factor for the refractive index is given by the expression

$$n^2 = (n - 1) \times \frac{1 - 0.00076 t'}{1 - 0.00076 t} + 1.$$

There is no generally agreed temperature which may be taken as a standard for the refractive indices of the fixed oils, and the author proposes a temperature of 40° C. as a standard temperature suitable both for oils and fats. Substituting 40° C. for t' in the above expression, the value of the refractive index of any fixed oil determined at any other temperature is calculated by the factor :

$$\frac{0.9696}{1 - 0.00076 t}$$

The error involved in using this method of correction is less the nearer the temperature of observation be to the standard temperature of 40° C., and it is better to

make the observations at any steady temperature near 40° C. than to maintain the instrument exactly at 40° C. The correction factor can then be used for recording the results at the standard temperature.

J. F. B.

Characterisation of Fuel Oils, particularly Petroleum Spirit, by the Picric Acid Value. J. Tausz and E. Schnabel. (*Chem. Zeit.*, 1919, 43, 726.)—The solubility of picric acid in the different types of hydrocarbons shows very wide divergencies, and may form the basis of a method for the approximate estimation of benzene in petroleum spirit. Picric acid is practically insoluble in *n*-hexane, but is soluble in benzene to the extent of about 10 per cent. About 50 grms. of the fuel to be examined are treated with excess of solid picric acid in a 200 c.c. stoppered bottle. About 3 grms. of the acid are initially added, and if this quantity proves to be insufficient a further quantity is added, so as to leave yellow crystals undissolved. The liquid is thoroughly shaken for ten minutes and then filtered. About 20 grms. of the filtrate are weighed out into a stoppered bottle, treated with 30 c.c. of water, and titrated with $\frac{N}{10}$ sodium hydroxide in the presence of phenolphthalein. The pink colour must persist after prolonged shaking. The picric acid value is expressed in terms of c.c. of $\frac{N}{1}$ alkali required per 100 grms. of the saturated solution. The picric acid value of light petroleum spirit (petrol) ranges between 0.05 and 0.3; the heavier fractions, which are used for lacquers, etc. ("white spirit"), show higher values—*e.g.*, 0.9 and 1.25; whereas crude 90 per cent. benzene has a picric acid value of 38.6, and commercial toluene, 43.0. In the case of mixtures the picric acid value is not a linear function of the percentage of benzene, but the delicacy of the method increases with the proportion of aromatic hydrocarbon present. The method is not very sensitive in the presence of less than 20 per cent. of benzene, but this circumstance is not of much importance, since genuine petroleum spirits contain a certain quantity of aromatic hydrocarbons, and the addition of further quantities of benzene to a natural product must always increase the picric acid value.

J. F. B.

Determination of the Saponification Value of Dark Resins. H. Salvaterra. (*Chem. Zeit.*, 1919, 43, 765-766.)—From 1 to 4 grms. of dark resins are saponified by boiling for thirty minutes beneath a reflux condenser with 50 c.c. of approximately $\frac{N}{2}$ alcoholic potassium hydroxide solution. The resulting soap solution is treated with 32 c.c. of approximately $\frac{N}{2}$ barium chloride solution and 300 c.c. of recently boiled neutral water, thoroughly shaken, and heated for three-quarters to one hour on the water-bath. It is then cooled, made up to definite volume (500 or 600 c.c.), and filtered, and 100 c.c. of the filtrate are titrated with $\frac{N}{2}$ hydrochloric acid, using phenolphthalein as indicator. In some cases the barium soap agglomerates and adheres to the bottom of the flask, rendering filtration before the titration unnecessary. At the same time a blank determination is made under the same conditions, in order to eliminate the error due to the unequal action of the atmospheric carbon dioxide upon the barium hydroxide, formed by interaction of the excess of the barium chloride and the potassium hydroxide.

C. A. M.

Estimation of Paraffins and Unsaturated Compounds in Commercial Toluols. H. G. Evans. (*J. Soc. Chem. Ind.*, 1919, 38, 402-405r.)—For the estimation of the paraffins in toluene the sulphonation method, carefully standardised, is the most convenient. About 50 c.c. of toluene are measured accurately to within 0.5 c.c. by pouring slowly upon the surface of 150 c.c. of 98 per cent. sulphuric acid contained in a 250 c.c. stoppered and graduated cylinder. The cylinder is shaken gently until fairly warm (not more than 45° C.), the pressure being released occasionally. After standing for about five minutes it is shaken vigorously for three or four minutes to complete the sulphonation. The contents are then transferred to the measuring vessel, consisting of a 350 c.c. separating funnel, the neck of which is fitted with a graduated tube reading to 0.01 c.c., and the stem of which is connected with a mercury reservoir. The last drops are rinsed in with 50 c.c. of sulphuric acid, the liquid is allowed to cool and finally the layer of paraffins is forced up into the graduated tube and measured. A correction must be applied for the solubility of the paraffins in the acid; this correction amounts to 0.60 per cent. on the volume of the toluene, to be added to the volume of the paraffins when this is below 5 per cent.; it is slightly higher, 0.62 to 0.63 per cent., for the range 5 to 10 per cent. of paraffins. An alternative method for the estimation of paraffins is based on the accurate determination of the sp. gr. at 15.5°/15.5° C. The sp. gr. of pure toluene at this temperature is 0.87193 and that of the paraffins separated by sulphonation is 0.73539, but, since the paraffins are always accompanied by a proportion of unsaturated compounds having a lower sp. gr. than pure toluene, the calculation of the percentage of paraffins must be made by using a corrected sp. gr. = 0.7208. In commercial toluenes the presence of 1 per cent. of paraffins by volume is associated with a lowering of the sp. gr. amounting to 0.00151, whereas in a synthetic mixture of pure toluene and separated paraffins the lowering is only 0.00136. Using the corrected figure, the results found by the specific gravity method are in good agreement with those by the sulphonation method, but a high degree of accuracy is necessary in the determination of the sp. gr., which must be correct to within 4 units in the fourth place of decimals. The method for the estimation of the unsaturated compounds in toluene consists in titrating with $\frac{N}{10}$ bromide-bromate solution in the presence of sulphuric acid: 10 c.c. of the sample are treated in a small stoppered bottle with 20 c.c. of dilute sulphuric acid (1:5) and the bromide-bromate solution, containing 9.9167 grms. of potassium bromide and 2.7833 grms. of potassium bromate per litre, is run in until the colour of the bromine persists in the toluene layer after five minutes' shaking. If the colour persists after standing for a further ten minutes the titration is considered to be finished. On an average, commercial toluene requires 9.58 c.c. of $\frac{N}{10}$ bromine solution, which corresponds to 0.2068 per cent. of unsaturated compounds as calculated from the sp. gr. In practical work, the estimation of the unsaturated compounds by bromide-bromate solution, and of the paraffins by sulphonation, gives a sufficiently accurate and rapid valuation of the sample.

J. F. B.

Criteria of the Degree of Purity of Commercial Toluene. J. S. Lumsden. (*J. Chem. Soc.*, 1919, 115 and 116, 1366-1372.)—Commercial toluene contains varying amounts of a liquid which cannot be nitrated and has distilled along with the toluene

during rectification. This liquid has not been isolated, but is probably of the nature of a paraffin mixture (B. Pt. 108-112. Density 0.743). The presence of 1 per cent. of such a liquid lowers the density of pure toluene (density 0.8712) by 0.0013, and a close approximation to the purity of a sample may be based upon a graph involving these data. *Estimation of Toluene by Nitration.*—Two methods may be adopted: (a) nitrating with a weighed excess of potassium nitrate, and determining the weight of nitric acid remaining: the amount of acid used is thus found which gives the weight of toluene nitrated; or (b) isolating and weighing the dinitro compound. The first of these works well, but with the second the whole of the dinitro compound cannot be recovered, as a small but definite trace remains in solution; this can, however, be allowed for. Examples of both methods, and the quantities used and obtained, are appended, together with working details. *Estimation of Toluene by Miscibility with Acetic Acid.*—Orton (*J. Chem. Soc.*, 1919, 115 and 116, 1055) has shown that when acetic acid of 88 to 90 per cent. purity is mixed with toluene in certain proportions two layers are formed, and, on raising the temperature, a point of complete miscibility is reached which is definite for the same strength of acid and so sharply defined that it can be read to $\frac{1}{10}$ degree. The method is accurate, but it is very difficult to prepare and keep strong acetic acid at a definite strength without absorption of moisture from the air, and the author has devised a modification which consists in the changing of an absolute to a comparative method which may be carried out in any commercial laboratory. A quantity of concentrated acetic acid of unknown strength is taken, and, with equal volumes of this, the temperatures of miscibility with equal volumes of pure toluene, toluene with 5 per cent. of paraffins, and the sample of toluene to be tested, are found. Then, since the percentage of paraffins is proportional to the increase in the temperature of miscibility, the comparison of the rise of temperature of miscibility of the sample with that of the 5 per cent. paraffin mixture gives the paraffin content of the sample. As the miscibility point is approached, the two layers disappear, the liquid becomes opalescent, and then suddenly transparent. This is the point at which the temperature is noted. The same procedure is adopted with the 95 per cent. toluene and with the sample under examination. It is not necessary that the strength of the acid should be known, so long as it remains constant during the time that the three portions are withdrawn. When the same samples of toluene were tested by the different methods, the following results were obtained:

From sp. gr. curve	...	...	92.3 per cent.
From nitrometer estimation after nitration	...	92.9	" "
By weighing dinitro compound	...	92.6	" "
By temperature of miscibility with acetic acid	...	92.5	" "

The author concludes that the sp. gr. of commercial toluene gives a fair indication of the amount of impurity present; the estimation of the temperature of miscibility with acetic acid is quickly done, and, although a comparative method, if skilfully carried out it is trustworthy; the nitration and estimation of the excess of nitric acid by the nitrometer are believed to be the most accurate test and to indicate the total nitration which has taken place, while the weighing of the solid after nitration gives the actual practical yield of nitro compound. By none of the methods, under ordinary conditions of working, can an accuracy closer than 0.5 per cent. be expected.

H. F. E. H.

INORGANIC ANALYSIS.

Rapid Estimation of Carbon Monoxide in Air. A. R. Lamb and A. T. Larson. (*J. Amer. Chem. Soc.*, 1919, 41, 1908-1921.)—Two rapid methods of estimating carbon monoxide in air are described. In the first method the mixture of gases is passed over a red-hot platinum wire, in contact with which the carbon monoxide is partially burned, and then through heated copper oxide to complete the combustion. After removal of the carbon monoxide, the gaseous mixture is conducted over a second platinum wire exactly similar in every respect to the first wire and heated by the same electric current. The first wire will be the hotter, owing to the combustion which occurs upon it, and this difference of temperature will cause a change in the relative resistance, which is indicated upon a suitable galvanometer. To insure uniformity of temperature in the surroundings the apparatus is enclosed in a regulated water thermostat, and the current of gas is conducted through long copper spirals, also within the thermostat, before reaching the platinum wires. It was found necessary to prevent eddies in the gas current, and this was effected by passing the gas through layers of cotton gauze before reaching the platinum wires. The second method is based upon the catalytic combustion of the gas at 275° to 300° C., in contact with platinum. The rise of temperature is measured by means of a thermo-couple and galvanometer, the deflections of the latter corresponding with definite concentrations of carbon monoxide. The apparatus consists of a vertical steel tube about 90 cm. long and 7.5 cm. in diameter, closed at one end, and covered, except for the uppermost 25 cm., with layers of asbestos paper. Diphenylamine is heated in this to produce a vapour-bath, the zone of condensation being kept within about 30 cm. from the top of the tube. The gaseous mixture is brought to the temperature of the vapour-bath by means of a 4 mm. pyrex glass tube extending to about 5 cm. from the bottom of the vapour-jacket, whence it winds backwards as a close coil about 15 cm. long and 6 cm. in diameter. Attached to this coil is a pyrex glass tube 15 mm. in diameter extending about 30 cm. above the top of the steel tube, and into this is fitted a tube containing the catalyst and thermo element. The catalyst is prepared from fine platinum wire or gauze. In the case of the former the wire is wound into a 2 mm. spiral and then compressed. The wire is cleaned with acid, then coated with platinum black electrolytically, washed, and electrolysed first in dilute sodium hydroxide solution and then in dilute sulphuric acid. The apparatus is calibrated by passing through it air containing known quantities of carbon monoxide at a constant speed and noting the deflections of the galvanometer. The introduction of halogens has a permanent toxic action upon the catalyst. Sulphur dioxide is also injurious, but its effect may be removed by raising the temperature to 350° C. and passing a current of air through the catalyst. It is shown that the method gives results accurate within 0.0038 per cent. on the total air mixture in the case of a mixture containing about 1 per cent. of carbon monoxide.

C. A. M.

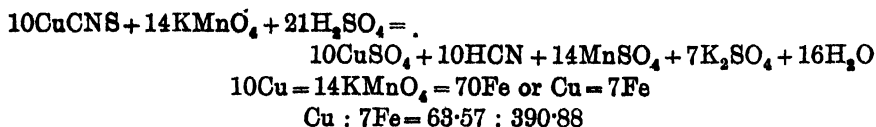
Rapid Tests of Cement. A Cavazzi. (*Annali Chim. Applic.*, 1919, 12, 59-73.)—Samples of cement alone and in admixture with 2 and 3 parts of sand

were exposed to the action of hot (100° C.) and cold soft and sea water for periods up to 140 days, and the usual compression and tensile tests then applied. The comparative results indicated that at first cement hardens much more rapidly in hot water, but that after the seventh day there is not so much difference between the hardening in hot and in cold water. The results obtained with twenty-five different slow-setting cements and four quick-setting cements in comparative tests after exposure of 7, 28, and 90 days in cold water, and 2, 4, and 6 days in hot water respectively, varied according to which periods were chosen for the comparison, but the general conclusion from the whole series of experiments is that the more rapid heat test yields results quite in accordance with practice. C. A. M.

Moisture in Blast Furnace Coke. W. H. George. (*J. Soc. Chem. Ind.*, 1919, 38, 394-396T.)—The best method of sampling when the wagon is unloaded through a side door is to wait until the wagon is one-third to one-half empty, and take the sample from the vertical sections thus exposed. The coke at the top is generally either wetter or drier than the rest owing to its exposure to the weather, and there is a tendency for the breeze (the wettest portion of the coke) to collect at the bottom during transit. Tests have shown that the small coke may contain two to eight times as much moisture as the large; further, the outer layer of a lump may contain ten times as much water as the interior. These points have to be borne in mind when selecting the samples, which should be taken at regular intervals apart; lumps should be broken through the middle. For routine work a sample weighing about 14 lb. consisting of about twenty pieces is sufficient. Owing to the extreme differences between the large coke and the small coke closer concordance than 1 per cent. is hardly to be expected. Crushing of the sample must be undertaken without delay, and the size of the sample must be reduced by quartering. Sieves should not be employed, as the loss of moisture during treatment is very rapid. The actual estimation of moisture in the prepared sample is a relatively simple matter, requiring none of the precautions which are necessary in the case of coal, and any temperature between 105° and 200° C. may be used. The distillation method is also applicable. The amount of water that can be absorbed by coke after immersion varies with its porosity and is given as 30.49 per cent. for hard coke, 39.26 per cent. for soft coke, and 51 per cent. for very soft coke. The effect of 1 inch of rain falling on a wagon designed to carry 12 tons of coke is to raise the moisture content of the coke by 2.4 per cent. J. F. B.

Permanganate Method for Copper. L. F. Clark. (*J. Ind. and Eng. Chem.*, 1919, 11, 1138-1139.)—The following modification of this method is recommended. The copper is precipitated as cuprous thiocyanate from acid solution in the usual manner. The filter-paper containing the cuprous thiocyanate is placed in the beaker in which the precipitation was made, 25 c.c. of hot 4 per cent. sodium hydroxide solution are added, and the mixture stirred until the paper is shredded and the precipitate decomposed. About 25 c.c. of cold 15 per cent. sulphuric acid containing 1 per cent. of ferric sulphate are next added. The result is a mixture of ferric thiocyanate, ferrous sulphate, cupric sulphate, and a large proportion of

cuprous thiocyanate re-forms when the mixture is acidified. The mixture is titrated with permanganate until the ferric thiocyanate colour gives way and the mixture becomes whiter. Concentrated hydrochloric acid (5 c.c.) is now added to aid the solution of the residual cuprous thiocyanate. Finally, the solution is titrated to a stable permanganate pink. The equation for the reaction may be written :



whence $\text{Cu} = 0.1626 \text{ Fe}$ (Fe value of the permanganate). This is the theoretical factor, and the actual factor under the conditions followed by the author is 0.1646, a closer agreement with the theoretical than is found in older modifications of the permanganate method. G. C. J.

Electrolytic Estimation of Copper in Pyrites and Pyrites Cinders.
E. Mengler. (*Chem. Zeit.*, 1919, 43, 729.)—The following method avoids the use of hydrogen sulphide to separate copper from iron. The pyrites (5 grms.) is treated with 50 c.c. of dilute (1 : 1) nitric acid, which is subsequently evaporated until fumes of sulphuric acid arise. The residue is redissolved in 50 c.c. of fuming hydrochloric acid, 2 grm. sodium hypophosphite dissolved in a little water are added, and the solution evaporated to dryness at 110-120° C., whereby all arsenic is reduced and got rid of. Cinders are dissolved in hydrochloric acid direct and evaporated as described. To convert the chlorides to sulphates, the residue is heated with sulphuric acid containing a little nitric acid, and heating continued until dense fumes arise. The nitric acid ensures the solution of copper sulphide which may be present as a consequence of secondary reactions during the preceding operations. The residue is taken up in hot water, the solution filtered off from the lead sulphate and insoluble matter, 1 gr. citric acid and 2 grms. ammonium nitrate are added, the solution is diluted to 200 c.c., and the copper deposited electrolytically, using a platinum cathode of 200 sq. cms. surface and a current of 0.3 to 0.4 ampère (2.8 volts). Not all the lead present is deposited on the anode; some comes down with the copper on the cathode, and the deposit must be redissolved in hot nitric acid and the solution re-electrolysed after cooling and addition of sulphuric acid. Pyrites free from arsenic and lead can be dissolved direct in 50 c.c. dilute (1 : 1) nitric acid and 20 c.c. dilute (1 : 2) sulphuric acid, evaporated to fuming, diluted, filtered, and electrolysed once after addition of citric acid and ammonium nitrate. The double precipitation in the process outlined above makes the process a slow one, but a number of estimations can be conducted simultaneously and the electrolysis can proceed overnight. The avoidance of the use of hydrogen sulphide is a point in favour of the method, which has been in use in the Duisburg copper works for twenty-five years. G. C. J.

Bibliography of the Use of "Cupferron" as a Quantitative Reagent.
S. A. Braley. (*J. Ind. and Eng. Chem.*, 1919, 11, 1144-1145.)—The author thinks

that "cupferron," the ammonium salt of nitrosophenylhydroxylamine (Baudisch: *ANALYST*, 1910, **35**, 78; 1911, 520), has not been given due consideration as a quantitative reagent. Reference is made to the works of Bellucci and Grassi (*ANALYST*, 1913, **38**, 390), Hanus and Soukup (*ibid.*, 1910, **35**, 453), R. Fresenius (*ibid.*, 1911, **36**, 175), Thornton (*ibid.*, 1914, **39**, 332, 565), Biltz and Hödke (*ibid.*, 1910, **35**, 327); and to that of James Brown (*J. Amer. Chem. Soc.*, 1917, **39**, 2359), who used the reagent to separate iron, titanium, and zirconium from manganese and aluminium; of Turner (*Am. J. Sci.*, 1916, [iv], **41**, 339) and Rodeja (*Anal. Soc. Espan. Fis. Quim.*, 1914, **12**, 379), who used it to separate vanadate from arsenate and phosphate; of Wissenson (*Zeitsch. anorg. Chem.*, 1911, **23**, 969), who used it to estimate iron in ferrocyanide; and of Schroeder (*Zeitsch. anorg. Chem.*, 1911, **72**, 94), who gives some additional data for the separation of iron from cobalt and nickel.

G. C. J.

Electrometric Titration. W. D. Treadwell and L. Weiss. (*Helv. Chim. Acta*, 1919, **2**, 680-697.)—The electrometric titration of silver (with sodium chloride), hydrogen sulphide (with silver), mercurous salts (with sodium chloride), cuprous salts (with thiocyanate), zinc (with ferrocyanide), ferrous salts (with dichromate), uranyl salts (with permanganate), vanadyl salts (with permanganate), and acidimetric estimations are trustworthy.

W. P. S.

Theory of Electrometric Titration. W. D. Treadwell. (*Helv. Chim. Acta*, 1919, **2**, 672-680.)—The author discusses the electrometric titration of silver, mainly from a mathematical point of view. The influence of the presence of other salts on the change in potential during the titration has been investigated.

W. P. S.

Indirect Method for the Electrolytic Estimation of the Halogens. J. H. Reedy. (*J. Amer. Chem. Soc.*, 1919, **41**, 1898-1902.)—Solutions of halogen salts may be electrolysed with silver anodes, with the formation of an adherent deposit of silver halide on the anode and without precipitation in the solution, provided that an anodic potential of 0.59 volt is not exceeded. The use of a 0.5 molar solution of sulphuric acid was found most suitable for maintaining the electrolytic conductivity of the solution of the halogen salt. The cathode used was of bright sheet platinum, whilst the anode was composed of a frame of heavy silver wire covered with fine silver gauze, the whole being coated electrolytically with silver, and subsequently heated in an electric oven to make the deposit firm and adherent. The deposit of silver halide upon this anode was weighed, and the electrode then made the cathode in a dilute solution of sodium hydroxide, which was electrolysed until hydrogen formed freely on the surface. The resulting deposit of silver was washed, and the electrode heated to about 500° C. in an electric furnace, and again weighed, the loss in weight on reduction representing the weight of the halogen. Even in the case of chlorine, where the potential required for complete deposition lies very close to the solution potential of silver, the latter does not begin to dissolve until the deposition of the halogen is practically complete. If the solution becomes opalescent it can be cleared again by lowering the potential below 0.59 volt.

C. A. M.

Electrolytic Separation of Lead from Chromium and the Analysis of Chrome Yellow and other similar Mineral Pigments. J. Milbauer and I. Setlik. (*J. prakt. Chem.*, 1919, **99**, 85; through *Chem. Zeit. Uebersicht*, 1919, **43**, 257.)

—Lead may be easily separated from chromium in the form of lead peroxide by using a solution containing 0.2 grm. of lead, 0.1 grm. of chromium, 20 c.c. of nitric acid (sp. gr. 1.4), and 15 c.c. of perchloric acid (sp. gr. 1.12) in 150 c.c. The mixture of nitric and perchloric acids readily dissolves all samples of lead chromate, including orange and red, on heating. On electrolysis of this solution, lead peroxide separates quantitatively and the chromic acid is reduced at the cathode to the chromium state. With a rapid rotation of the cathode (800 revs. per minute) and a current of 3.5 ampères at 2.5 volts, the separation of the lead peroxide and the reduction of the chromic acid is completed in half an hour.

J. F. B.

Application of Rotating Reductors in Estimation of Iron. W. Scott. (*J. Ind. and Eng. Chem.*, 1919, **11**, 1135-1137.)—Ferric sulphate is rapidly reduced by rapidly rotating reductors of zinc or aluminium, either with or without the aid of a current of electricity. Quantities of iron such as 70 mgrms. are reduced quantitatively in less than three minutes by the use of a zinc reductor of 45 sq. cm. surface making 800 revs. per minute. For the reduction of such small quantities, the use of a current presents no advantage. In the reduction of quantities like 700 mgrms., using a rotating zinc cylinder of 75 sq. cms., the time required may be reduced slightly by using a current of 6 ampères, but by working at a boiling temperature only four minutes are required even without the use of a current. Aluminium operates somewhat more rapidly than zinc, but there is greater difficulty in obtaining aluminium of the necessary degree of purity.

G. C. J.

Detection of Molybdenum by Means of Xanthic Acid. J. Koppel. (*Chem. Zeit.*, 1919, **43**, 777-778.)—This reaction, first noted by Siewert in 1864 (*Zeitsch. ges. Naturw.*, 1864, **23**, 5), has recently been referred to by Malowan (*Zeitsch. anorg. Chem.*, 1919, **108**, 73) as having been entirely overlooked by compilers of textbooks. It is, however, to be found in Gmelin-Kraut's *Handbuch* (7 Aufl., Band III., Abt. 1, 907), from which the author became acquainted with it many years ago. He adds a few lumps of potassium xanthate to the cold solution of the molybdenum compound, which solution should be as nearly as possible neutral. Mineral acid is then added. A yellow opalescence, due to separated xanthic acid, first appears, but in presence of molybdenum this becomes gradually red, and finally deep blue. If very large quantities of molybdenum are present, black oily drops separate. Malowan says the reaction is only produced by freshly prepared solutions of potassium xanthate, and that the solid reagent is useless. The author agrees that solutions do not keep, but finds the solid quite satisfactory. Oxalates must be absent, as their presence enormously reduces the sensitiveness of the reaction which will detect 0.005 mgrm. molybdenum in solutions containing as little as 0.0007 per cent. The fact that the coloured compound is soluble in chloroform makes it possible to detect molybdenum in even lower concentrations. Copper, cobalt, nickel, iron, uranium, and tungsten interfere.

G. C. J.

Volumetric Estimation of Nickel. J. Holluta. (*Monatsh. Chem.*, 1919, 40, 281-291; through *J. Soc. Chem. Ind.*, 1919, 38, 965A.)—The process depends on the fact that when a nickel salt reacts with dimethylglyoxime, acid is liberated, which is estimated by titration with alkali in the presence of phenolphthalein or methyl red. A standard $\frac{N}{50}$ solution is prepared by dissolving 4.64 grms. of dimethylglyoxime in the requisite quantity of 97 per cent. alcohol (300 to 400 c.c.); 20 c.c. of $\frac{N}{1}$ potassium hydroxide solution are added with constant shaking, the solution is diluted to 1,000 c.c. with CO_2 -free distilled water, and filtered after twenty-four hours from any slight precipitate of potassium carbonate (the alcohol used must be free from aldehyde and neutral in reaction). The alkali content of the solution is checked by titration with standard acid, using phenolphthalein or methyl red as indicator. The nickel solution under examination is diluted to the required extent, exactly neutralised with $\frac{N}{10}$ or $\frac{N}{20}$ potassium hydroxide solution, 2 to 5 c.c. of the standard dimethylglyoxime solution are added, and, after vigorous agitation, the mixture is gently warmed, whereby the precipitate is caused to collect, leaving a clear liquor; the standard solution is then gradually added with frequent agitation until the solution acquires a permanent pale pink coloration. In consequence of the bulky nature of the nickel precipitate the amount of the metal in the solution should not exceed 0.03 grm. The method is particularly advantageous in dealing with very small quantities of nickel, and titration can readily be effected with $\frac{N}{100}$ solutions if methyl red is used as indicator. The results are not affected by the presence of the alkali salts of strong acids.

Method for the Analysis of Nitrous Gases and its Application to the Study of Towers for the Absorption of such Gases. A. Geake and F. J. Squire. (*J. Soc. Chem. Ind.*, 1919, 38, 387-369T.)—The method described was devised for the analysis of gases in a system of absorption towers used in connection with a plant for the denitration of nitroglycerin spent acid; the gases consisted of a mixture of nitric oxide and peroxide, carbon monoxide and dioxide, oxygen and nitrogen. The sample of gas was collected in a bottle of such capacity that the sample contained about 0.3 grm. of nitrogen oxides; this bottle was fitted with a stopper through which passed a tapped tube. The portion of the tube inside the bottle was bent so as to reach nearly to the side of the bottle near the shoulder. The bottle was charged with 25 c.c. of $\frac{N}{2}$ permanganate solution and 1 c.c. of sulphuric acid, and the air was then exhausted as completely as possible. To take the sample of gas the external part of the tube (this had a diameter of 2 to 3 mm.) was inserted in the sampling hole while the bottle was held in an inclined position so that the permanganate solution covered the inner end of the tube, and the tap was opened for about one second, closed, and the bottle shaken thoroughly. The residual volume of gas, still under reduced pressure, was then measured by connecting the bottle with an Orsat apparatus. The portion of gas passing into the Orsat apparatus was used for the estimation of carbon dioxide (by sodium hydroxide) and oxygen (by phosphorus); the residue was taken to be pure nitrogen. Actually it contained carbon monoxide, but, as this is inactive, and has the same density as nitrogen, its presence was unimportant. Air was now admitted to the sample bottle,

and the excess of permanganate titrated with $\frac{N}{2}$ ferrous sulphate solution. The total combined nitrogen was estimated in this titrated solution by the ferrous sulphate method described by Bowman and Scott (ANALYST, 1915, 40, 474). All the calculations were made in weights, the unit taken being 1 lb. per hour, and an explanation is given of the methods of making these calculations of the weights of the constituent gases passing through the plant.

W. P. S.

Estimation of Titanium in certain Italian Puzzuolanas. A. Cavazzi. (*Annali Chim. Applic.*, 1919, 12, 105-111.)—For the estimation of titanium in puzzuolanas the sample is finely powdered in an agate mortar, and 0.5 gm. moistened with 20 drops of dilute (1:1) sulphuric acid in a platinum crucible, mixed with 10 c.c. of hydrofluoric acid, and the liquid evaporated to dryness. The residue is heated with sulphuric acid to carbonise organic matter, then pulverised and fused with 4 grms. of potassium pyrosulphate at a temperature insufficient to liberate fumes of sulphuric acid, until the mass becomes of a grey tint. The temperature is then increased until the residue becomes yellowish-white, and is finally heated over a blow-pipe flame until the mass has become reddish-orange, but without excessive fumes appearing. The fused mass is dissolved in water and the solution, made up to 100 c.c., is stirred at intervals for thirty minutes and filtered. The titanium in the filtrate is estimated by adding 2 c.c. of 3 per cent. hydrogen peroxide and 2 c.c. of sulphuric acid and comparing the coloration with that given under the same conditions by standard solutions of titanium. A convenient method of preparing the standard solution is to use titanium sulphate, the amount of titanium oxide (TiO_2) in which may be rapidly estimated by heating the salt with successive quantities of ammonium carbonate until all sulphuric acid has been removed, and weighing the residue. The same method may also be used with any salt of titanium which is soluble in sulphuric acid and can be converted into pure TiO_2 by ignition. The amount of titanium estimated by this colorimetric method in four samples of Italian puzzuolanas ranged from 0.312 to 0.558 per cent.

C. A. M.

New Colorimetric Method for Estimation of Uranium. Von Müller. (*Chem. Zeit.*, 1919, 43, 739-740.)—The author finds that uranyl salts give intense colorations with aliphatic α -hydroxy and ketonic acids, with aromatic hydroxy-acids, and especially with polyhydric phenols. The colorations given by the polyhydric phenols are the most intense, but these substances are not suitable for quantitative colorimetric work because they and their compounds are so readily oxidised with consequent change in the intensity of the coloured compounds. Salicylic acid is free from this objection, and may be used for the colorimetric estimation of uranium. As little as 0.01 per cent. can be detected, and the most convenient concentration for colour comparisons is 0.05 per cent. uranium. The uranyl solution, which must be free from iron and from mineral acids and must not contain large quantities of free acetic acid, is mixed with an equal bulk of a 2 per cent. solution of sodium salicylate, and comparison is made with a standard solution in a Dubosc colorimeter.

G. C. J.

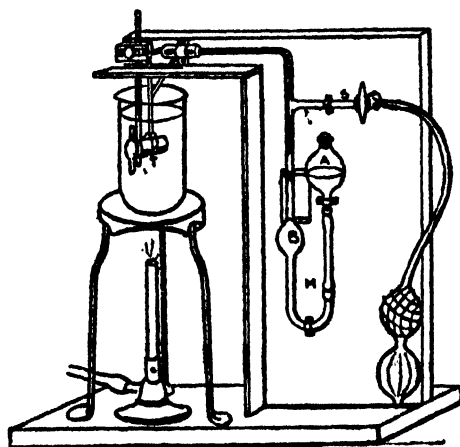
Analysis of Vanadium Compounds by Means of Carbon Tetrachloride. P. Jannasch and H. E. Harwood. (*J. prakt. Chem.*, 1918, 97, 98; through *Chem. Zeit. Übersicht*, 1919, 43, 209.)—A general method for the separation of vanadium from other metals depends on the volatility of vanadium oxychloride when the vanadium salt or compound is heated in a current of carbon tetrachloride vapour. The carbon tetrachloride is added drop by drop to sulphuric acid heated at 120° to 140° C., and the vapour is led into the slightly heated tube containing the vanadium compound. A current of carbon dioxide is also passed through the tube, and, when all the vanadium has been volatilised, the residue is allowed to cool in a current of this gas. The vanadium oxychloride is collected in a receiver containing 200 c.c. of water and 50 c.c. of nitric acid. The contents of the receiver are then evaporated, the vanadic acid dissolved in dilute sulphuric acid, reduced with sulphurous acid, and the resulting bright blue vanadyl sulphate solution titrated with hot permanganate solution.

W. P. S.

APPARATUS, ETC.

Determination of the Softening-Point of Asphaltum and other Plastic Substances. D. F. Twiss and E. A. Murphy. (*J. Soc. Chem. Ind.*, 1919, 38, 405-406r.)—The apparatus illustrated in the figure has been designed to overcome

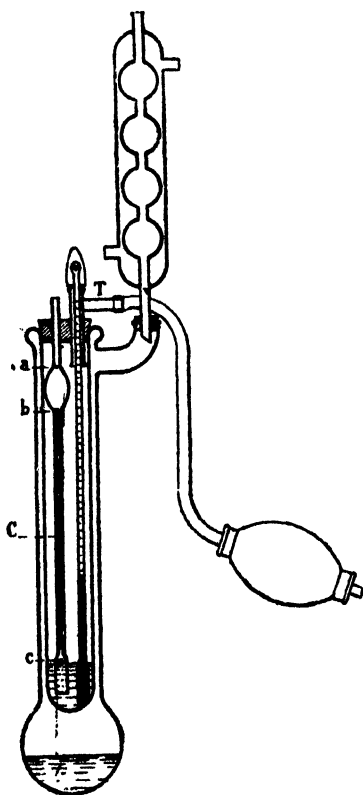
certain objections attendant upon the Krämer-Sarnow method, which is regarded as the most satisfactory method hitherto proposed. The apparatus consists of a U-tube, *M*, one arm of which is connected by capillary tubing with a gun-metal tap, *T*. The central plug of this tap has a conical or tapered bore, $\frac{1}{4}$ in. in length and $\frac{1}{8}$ in. and $\frac{1}{16}$ in. in diameter at the respective ends. The bore is terminated at each end by a flat groove, to which its axis is perpendicular; the capillary metal tube of the tap is connected with the glass capillary by means of a well-sealed metal sleeve. To carry out the test



the sample of bituminous material and the clean plug of tap *T* are warmed in a steam-oven for about ten minutes so that the material becomes somewhat plastic; a piece of the softened material is then pressed into the wider end of the bore of the warm tap plug as speedily as possible with a small spatula until the bore is filled and a little protrudes from the other end. The tap plug is then allowed to cool and the excess of material is carefully removed so as to leave the exposed surfaces of material flush with the metal. The plug is refitted in the tap with the narrow end of the bore facing downward. Air is forced into the apparatus until the

U-tube containing mercury indicates an internal pressure equivalent to 1.5 in. of mercury. The medium in the bath is then warmed until the bitumen in the tap plug is so softened that it is forced completely through the narrow end of the bore, a point which is immediately indicated by the mercury in the U-tube. The construction of the tap in metal affords ready transmission of heat, and, since the indication of the softening-point takes place outside the bath, any discoloration of the latter is of no account. The results obtained with this apparatus are substantially higher than by the Krümer-Sarnow method, the differences ranging from 5° to 30° C. The apparatus is equally suitable for the examination of balata and gutta-percha. It is essential that the material to be examined be prepared free from air and moisture.

J. F. B.



New Form of Viscometer. G. Baume and H. Vigneron. (*Ann. Chim. anal. appl.*, 1919, 1, 379-383.)—The apparatus here shown affords a simple means of determining the viscosity of liquids either in terms of absolute viscosity or in conventional degrees (Barbey or Engler). The viscosimetric tube, *C*, of the capillary type, is immersed at one end in the oil, etc., under examination in the tube *C*, whilst its upper end passes through the rubber stopper of *C*. This tube is surrounded by an outer thermostatic jacket in which a liquid of constant boiling-point (ether, 33.5° C.; acetone, 56.5° C.; benzene, 80.3° C.; or water) is kept boiling, the upper branch of this tube being connected with a reflux condenser. After the thermostatic liquid has boiled for fifteen minutes, oil is forced up into the viscosimetric tube by compressing the bulb connected with *T*. The time in seconds occupied by the oil in falling between the points *a* and *b* is noted, and the absolute viscosity is calculated by means of the formula $\eta = K.d.t$, where *K* represents a constant of the apparatus, *d* the sp. gr. of the liquid, and *t* the time. Duplicate determinations should agree within a few tenths of a second. The relationship between the absolute viscosity and the Barbey and

Engler degrees is found by means of the formulæ,

$$E = \frac{668}{F}, \quad E = \frac{\eta}{0.0732d}$$

where *E* and *F* represent the respective Engler and Barbey degrees, η the absolute viscosity, and *d* the sp. gr. of the liquid.

C. A. M.

Clip for preventing Rubber Connections from slipping off Glass and Metal Tubing, and for attaching Pressure Pumps to Taps. C. A. Keane and G. Patchin. (*J. Soc. Chem. Ind.*, 1919, 38, 391r.)—The clip is made in two halves hinged together and held in the closed position by means of a thumbscrew so that it encloses the rubber connection around the glass tube. For attaching a pump to a water tap a special form of hinged clip provided with a sleeve is employed. The tap and pump are first connected with rubber tubing, and the clip with the sleeve is fitted over the rubber attached to the pump. A second hinged clip without a sleeve is then clamped over the portion of the rubber attached to the tap. The second clip should slightly overlap the top of the sleeve of the first one. This form of attachment prevents the bulging of the rubber when the pump is in use. J. F. B.

New Method for the Physico-Chemical Analysis of Precipitates. Application to the Study of Calcium Phosphates. P. Jolibois. (*Comptes rend.*, 1919, 169, 1161-1163.)—The reacting solutions are allowed to mix in the stem of a Y-tube, to which they are admitted separately through the two arms; the solutions mix in about 0.01 second. The author has studied the reactions taking place when calcium hydroxide solution (1 gm. of CaO per litre) is mixed with phosphoric acid solutions of varying strength, the precipitates formed after one second being separated and analysed. Dicalcium phosphate forms only in a solution containing at least 0.800 gm. of P_2O_5 per litre in the form of monocalcium phosphate, whilst tricalcium phosphate is stable only in a solution containing less than 0.1 gm. of P_2O_5 per litre as monocalcium phosphate. A well-crystallised phosphate having the formula $P_2O_5 \cdot 2CaO, P_2O_5 \cdot 3CaO, 10H_2O$ was obtained when the solution contained from 0.375 to 0.870 gm. of P_2O_5 per litre as monocalcium phosphate; if the P_2O_5 content of the solution was increased this compound changed gradually into dicalcium phosphate. W. P. S.



REVIEWS.

AN INTRODUCTION TO THEORETICAL AND APPLIED COLLOID CHEMISTRY. By Wolfgang Ostwald: translated by Martin H. Fischer. Pp. xv+232. New York: John Wiley and Sons. London: Chapman and Hall. 1919. Price 11s. 6d. net.

The last twenty years have witnessed remarkably successful attempts to bridge the gap existing between molecular solutions on the one hand and coarse mechanical suspensions on the other. The author speaks of this gap as "the world of neglected dimensions," and in his enthusiastic exposition refers to colloid chemistry as a separate and independent science. "Since the birth," he says, "of the so-called classical physical chemistry of the molecular solutions, some thirty years ago, no branch of physics or chemistry has arisen which can be compared in importance, so far as scientific and technical applications are concerned, with that of colloid chemistry." Whether this contention is justified or not, the author has produced in the present volume an authoritative summary of the physics and chemistry of dispersed systems which is well worth perusal by chemists of all kinds.

The book is based on a series of lectures delivered in North America in the winter of 1913-14, the lectures being designed mainly for audiences without any specialised knowledge of colloid chemistry. The survey is therefore a general one, but is at the same time very comprehensive, as may be seen from the main headings of the five chapters: Fundamental Properties of the Colloid State; Colloids as Examples of Dispersed Systems; Methods of Preparing Colloid Solutions; Classification of the Colloids: The Physico-chemical Properties of the Colloids and their Dependence on the Degree of Dispersion; The Changes in State of Colloids; Some Scientific and Technical Applications of Colloid Chemistry.

So numerous are the points at which the author detects the trail of the colloid that organic and analytical chemists, scientific and technical workers, will all find interest and stimulus. In the region of analytical chemistry reference may be made, for example, to the passing of precipitates through filters, to the Cassius purple test for gold, to Donau's borax bead method of detecting minute traces of the noble metals, and to Ley's test for distinguishing natural from artificial honey, as cases where the principles of colloid chemistry are applicable.

The author discusses at some length, and rightly so, the exact meaning to be attached to the term "colloid," and the relation of colloids to mechanical suspensions on the one hand, and to molecular solutions on the other. Colloids, it must be clearly borne in mind, are not a particular class of chemicals, for apparently every substance may assume the colloidal state under suitable conditions. The colloidal state is determined solely by the degree of dispersion, and colloids are accordingly defined as dispersed systems in which the diameter of the dispersed particles in typical cases lies between $0.1\ \mu$ and $0.001\ \mu$. This clear definition suggests criticism of the author's introduction of the term "isocolloid," by which he appears to indicate certain substances capable of furnishing both the dispersion medium and the dispersion phase, and which, therefore, seems to the reviewer to be incongruous with "colloid."

The relation of colloid chemistry to the older branches of the science is, in various directions, somewhat ill defined, and one prominent feature of the present volume is the challenging attitude adopted towards some of the generally accepted views held by physical chemists. The author tilts, for example, at the rôle assigned to osmotic pressure in connection with water movement in organisms, at the neglect of the capillary factor in the theory of freezing-point depression, and at the van't Hoff theory of solid solution and the phase rule as applied to alloys in which the degree of dispersion is particularly high. Such pleas for the reconsideration of current views rather add to the stimulating character of the volume, but it is unlikely that the author's contentions will be satisfactorily established unless colloid chemistry can be developed, much more than has been found possible hitherto, on the quantitative lines to which physical chemistry owes the strength of its present position.

JAMES C. PHILIP.

THE LIBRARY OF THE CHEMICAL SOCIETY. A RECORD OF A RECENT ATTEMPT AT CO-OPERATION. By F. W. Clifford. Reprinted from the Library Association Record, 1919, Caxton House, London.

In this reprint from the Library Association Record, Mr. Clifford gives an account of the inception of the scheme inaugurated two years ago for widening the scope of the Chemical Society's library by adding to it works of a technical character hitherto regarded as being outside the range of that Society's purview.

The outstanding feature of the scheme, however, is the extension of the valuable privilege of using the library to the members of other societies which in their corporate capacity have arranged to give it financial support. These are the Association of British Chemical Manufacturers, the Biochemical Society, the Faraday Society, the Institute of Chemistry, the Society of Chemical Industry, the Society of Dyers and Colourists, and the Society of Public Analysts. The members of these, whether Fellows of the Chemical Society or not, are allowed the full use of the library both for loan and reference, and the value of the privilege is increased by the fact that the hours of opening have been extended, being now from 10 a.m. to 9 p.m.; Saturdays, 10 to 5.

That stern taskmaster, the Great War, is responsible for bringing about this far-reaching change in the attitude of the oldest of the societies connected with chemical science, and, as a first step towards the removal of the arbitrary barriers which have hitherto separated the pure and the applied branches of the science, can only be to the good. It is greatly to be hoped that it will not stop here, but will eventually lead to a much closer association between some of the societies concerned.

One thing seems quite clear—namely, that with the extended use of the library and the addition to its shelves of books dealing with fresh classes of subjects, the premises at Burlington House will soon become as hopelessly inadequate, as they always were unsuitable, for their purpose.

A building consisting mainly of staircase with a library perched at the top, so small that many books have to be stored in the basement forty or more feet below, and unprovided with a lift, can hardly be considered a fitting home for the premier chemical society, or worthy of the science to which the country owes so

much. In recognition of its services during the war, surely the Government might find some edifice—even a *small* hotel might do—where all the societies concerned with the science could have a common meeting-place, with ample space for the library and a museum of old apparatus (why not modern, too?) and prints and pictures connected with the early days of the science. The apathy of the chemist towards the historical side of his subject is notorious, and the appliances of the earlier workers are in many cases either left to decay or find their way to America, where the great Universities seem to be more alive to interest of these relics of bygone days.

There is one other matter which has long been talked about which the broad-minded policy of the Chemical Society must surely have brought at last within the range of practical politics. It is a standing reproach to our commonsense that four or five different journals should at the same time be abstracting the same papers and printing them each in different type and on pages of a different size, when, with co-operation of the various institutions concerned, much of all this unnecessary labour and expense might be avoided. No doubt there are serious difficulties in the way—vested interests might have to suffer and long-established practices have to be given up—but the latest enterprise of the Chemical Society warrants the belief that even these obstacles might be overcome.

C. H. CRIBB.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

ANNUAL GENERAL MEETING AND ORDINARY MEETING, HELD
FEBRUARY 4, 1920.

The Annual General Meeting of the Society was held on Wednesday, February 4, in the Chemical Society's Rooms, Burlington House, W. The President, Dr. S. Rideal, occupied the chair.

The minutes of the previous annual general meeting were read and confirmed.

Messrs. T. Macara and C. Revis were appointed scrutators of the ballot-papers for election of Officers and Council for 1920.

The Acting Honorary Treasurer presented the accounts of the Society for 1919, and votes of thanks were passed to the Acting Hon. Treasurer and Hon. Secretaries.

Messrs. Marreco, Houseman, and Rixon, chartered accountants, were appointed auditors of the Society's accounts for 1920.

The President delivered his annual address.

Mr. Otto Hehner proposed that a hearty vote of thanks be accorded to the President for his address and for his services in the Chair, and that his permission be asked to print the address in THE ANALYST. This was seconded by Mr. G. N. Huntly, and the motion was carried.

The scrutators having reported the result of their examination of the ballot-papers, the President announced that the Officers and Council for 1920 had been elected in accordance with the Council's nominations, as follows:

President: Alfred Smetham.

Past-Presidents Serving on the Council: Messrs. Leonard Archbutt, Edward J. Bevan, A. Chaston Chapman, Bernard Dyer, Otto Hehner, Samuel Rideal, E. W. Voelcker, J. Augustus Voelcker.

Vice-Presidents: W. J. A. Butterfield, G. N. Huntly, G. R. Thompson.

Hon. Treasurer: Edward Hinks.

Acting Hon. Treasurer: E. W. Voelcker.

Hon. Secretaries : P. A. Ellis Richards, E. Richards Bolton.

Other Members of the Council : Messrs. F. W. F. Arnaud, W. Bacon, F. H. Carr, H. G. Colman, G. D. Elsdon, P. J. Fryer, R. Hellon, H. Hurst, C. A. Keane, S. E. Melling, G. W. Monier-Williams, W. Lincoln Sutton.

An ordinary meeting followed the annual meeting, the newly-elected President, Mr. Alfred Smetham, F.I.C., being in the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates were read for the first time in favour of Messrs. Herbert John Evans, B.Sc., F.I.C., Cornelius Durham Garbutt, Henry Atlas, John Carmichael, F.I.C., Maurice S. Hutchinson, B.Sc., Geoffrey Martin, D.Sc., Ph.D., F.I.C., Robert Leitch Morris, F.I.C., James Wood, M.A., B.Sc., F.I.C., Douglas Henville, F.I.C., and Benjamin Richards James, F.I.C.

A certificate was read for the second time in favour of Mr. William John Read, M.Sc., F.I.C.

The following were elected Members of the Society: Messrs. Percy Ewart Bowles, F.I.C., Ph.D. (Heidelberg), Charles Crowther, M.A. (Oxon), Ph.D. (Leipsic), John William Hawley, B.Sc. (Lond.), A.I.C., Ernest Joseph Lush, B.A. (Cantab.), A.I.C., H. Percy Marks, B.Sc., A.I.C., Robert Selby Morrell, M.A. (Cantab.), Ph.D. (Würzburg), F.I.C.

The following papers were read: "An Investigation into the Composition of the Unsaturated Hydrocarbons present in Coal Gas," by F. S. Sinnatt, M.B.E., M.Sc., F.I.C., and L. Slater, M.Sc., A.I.C.; "The Estimation of the Available Oxygen in Sodium Perborate and in Perborate Soap Powders," by H. Trickett, F.I.C.



OBITUARY.

WE deeply regret to record the death on February 7, 1920, of Mr. W. W. Fisher, M.A., F.I.C., a past President of the Society. An obituary notice will appear in a subsequent issue of the ANALYST.



ANNUAL ADDRESS OF THE RETIRING PRESIDENT.

(Delivered at the Annual General Meeting, February 4, 1920.)

THIS is my valedictory address as your President, and it brings forty-four years of the Society's existence and of the ANALYST to a close. It coincides with the passing of the Local Government Board and the birth of the Ministry of Health.

We began in 1875, a few years after the European struggle of 1870, and are now awaiting the blessings of the new peace which is gradually emerging from, let us hope, the last of these deadly international conflicts.

During the year our Society has progressed, our total number of members to December 31, 1919, including eleven honorary members, is 448 as against 439 for the previous year. We have welcomed twenty-four new members, and have to record the deaths of seven; whilst there have been six resignations from our ranks, and two members have been removed for non-payment of subscription.

The names of the deceased members are: Sir William Crookes (honorary member), John Richard Brooke, Thomas Fairley, Sidney Harvey, George Pilkington, Sir Boverton Redwood, John Charles Umney, and Edward G. Love (New York).

Sir William Crookes was one of the honorary members, and his life work is of world-wide recognition. He published his "Select Methods in Chemical Analysis" in 1871, and stated in his Preface to that work that he had tested most of the processes which had appeared during the previous twelve years in the *Chemical News*, and as some of them had proved of great value, it was thought that a service would be rendered to analytical chemistry if these trustworthy methods of analysis were systematically arranged in a convenient form for laboratory use. There have been few laboratory books which have survived for fifty years. Sir William remarked in 1871 that it was strange that *modern works* on analysis ignored about twenty of the elements, and that even Fresenius at that time only gave a separate form for their detection.

Obituary notices have appeared, in the ANALYST, of Thomas Fairley of Leeds, at one time in this chair as President of the Society, and of Sidney Harvey, a former Vice-President, who also took an active interest in our proceedings. Sir Boverton Redwood was well known in the technology of petroleum, and during his life advanced our knowledge of the methods of testing petroleum and its products.

John Charles Umney, like Sir Boverton Redwood, owed his connection with our Society to his father's pharmaceutical interests, and gave a considerable time to the study of the essential oils.

John Richard Brooke, a Government analyst in Singapore, and George Pilkington were Fellows of the Institute of Chemistry, the latter joining our Society in 1892.

During the session twenty-four papers were read at our meetings, and thirty-two original communications and twenty-seven "Notes" on analytical and kindred matters have been published in the ANALYST. The number of pages is the same as last year—viz., 428.

Titles of papers published in the ANALYST during the year 1919:

- "Estimation of Cacao Shell." By A. Knapp and B. G. McLellan.
- "Estimation of Phenacetin and other Para-Aminophenol Derivatives by Hypochlorous Acid." By A. D. Powell.
- "Effect of Morphine Concentration on the B.P. Method of Morphine Estimation." By H. E. Annett and Hadayal Singh.
- "Methods of Calculating Added Water in Milk." By L. Harris.
- "Fat Extraction Apparatus." By E. Griffiths-Jones.
- "An Instrument for the Determination of Small Quantities of Carbon Monoxide in Hydrogen." By E. K. Rideal and H. S. Taylor.
- "The Use of Ortho-Tolidine as a Colorimetric Test for Gold." By W. B. Pollard.
- "Regeneration of Nessler Solution." By D. Pullman.
- "The Estimation of the Approximate Quantity of Meat in Sausages and Meat Pastes." By G. Stubbs and A. More.
- "The Analysis of Sausages, Meat Pastes, and Army Rations." By A. Stokes.
- "A Combined Method for the Estimation of Small Quantities of Acetone, Alcohol, and Benzene in Air." By Major Elliot and J. Dalton.
- "The Electrical Conductivity of Milk." By J. H. Coste and E. T. Shelbourn.
- "Notes on Soluble Lead in the Glaze of Casseroles." By H. Masters.
- "The Detection and Estimation of Cocaine, Heroine, and Veronal in Viscera." By P. A. Ellis Richards.
- "The Estimation of Small Quantities of Antimony." By W. Beam and G. A. Freak.
- "Estimation of Small Quantities of Lead in Foods and Substances containing Calcium Phosphate." By B. W. J. Warren.
- "Milk Calculations: A Criticism." By H. Droop Richmond.
- "A Method for the Determination of Monochlorobenzene in Mixtures containing Benzene, Monochlorobenzene, and Dichlorobenzene." By N. G. S. Coppin and F. Holt.
- "The Composition of Buttermilk." By T. R. Hodgson.
- "Note on the Oil of *Ceratotheca Sesamoides*." By E. R. Bolton.
- "Studies in Steam Distillation—Part VI.: The Possibilities and Limitations of Duclaux's Methods for the Estimation of Homologous Acids." By H. Droop Richmond.
- "A Rapid Method for Determining Nickel and Cobalt in Ores and Alloys," Part III. By W. R. Schoeller.
- "An Improved Method for the Estimation of Nitrates in Water by Means of the Phenolsulphonic Acid Reaction." By Robert C. Frederick.
- "Mexican Insects in Poultry Food: Mexican Cantharides, *Notonecta*, *Corixa*, and *Berosus*." By T. E. Wallis.
- "Rapid Method for the Estimation of Titanium in Titaniferous Iron Ores." By John Waddell.
- "The Necessity for a Supply of Pure Agricultural Lime." By G. S. Robertson.
- "Milk Calculations: A Reply." By L. J. Harris.
- "Estimation of Morphine in Indian Opium." By Jitendra Nath Rakshi and Frank J. D'Costa.

- "The Examination of Commercial Samples of Nicotine." By Percival J. Fryer and C. H. Fryer.
- "The Analysis of Brazilian Zirconium Ore." By A. H. Powell and W. R. Schoeller.
- "The Halogen Absorption of Turpentine." By Ethel M. Taylor.
- "Egyptian Bricks." By G. Rudd Thompson.

Three papers emanated from the Ministry of Munitions, four were from India and the Colonies. The paper on the Estimation of Cacao Shell and the two papers on Sausages and Meat Pastes had their origin in attempts made by our members to assist the Ministry of Food to make Food Orders on these subjects workable.

We reviewed twenty-seven new books in the year as against thirty-one in 1918.

I am sure you will agree with me in thanking our editor and his abstracting staff for producing such a readable volume, especially when I understand that the sales of the ANALYST at the increased rate of subscription have been maintained, and that we have therefore a satisfactory balance-sheet from this source. The Publication Committee have already reported to the Council of the Society their satisfaction at the results of this change.

Our Treasurer is at last to be relieved of his war duties. I understand a cable has been received saying that he left Basrah on January 15 in H.M.S. *Rheinfels*, and I am sure we shall all welcome him back.

Our relations with the Institute of Chemistry continue to be of a friendly character. The joint circular letter of January, 1919, sent out by Sir Herbert Jackson and myself to the Clerks of the County Councils, City and Borough Councils concerned with the administration of the Sale of Food and Drugs Acts, and the Fertilizers and Feeding Stuffs Acts, brought in a number of replies which showed that many of the authorities were aware of the peculiar disadvantages which analysts under these Acts were suffering owing to the considerable increase of cost for laboratory work. I am sorry, however, to report that we have still not heard from quite a large number of districts, and it was consequently desirable that a further letter should be sent out again this year. This letter, dated January 16, 1920, was as follows:

"DEAR SIR,

"We are desired by the Councils of the Institute of Chemistry and the Society of Public Analysts to direct the attention of authorities concerned with the administration of the Sale of Food and Drugs and the Fertilizers and Feeding Stuffs Acts to the position, under the prevailing economic conditions, of Public Analysts and Official Agricultural Analysts.

"An impression appears to prevail rather widely that the salaries and emoluments allowed by the authorities are wholly in the nature of profit to the analysts; but this is far from being the case, as the analysts have to meet the cost of maintaining their laboratories and staffs, to pay higher salaries than formerly to their assistants,

and to bear the greatly increased cost of materials and apparatus in addition to the increased cost of living. The salaries of qualified assistants are generally more than double, and the prices for apparatus are about three times, those paid before the war.

"The Councils of the Institute of Chemistry and the Society of Public Analysts understand that some authorities have already taken the prevailing conditions into consideration, and have revised the terms of their analysts' appointments; but the Councils learn that only in very few instances have the analysts' salaries or fees been adequately advanced, while more generous consideration has been given to non-technical officers and employees, whose positions, although they do not necessarily involve the provision of staff and equipment at their own cost, have been similarly affected by the decreasing value of money.

"We may remind you that, under the provisions of the Sale of Food and Drugs Acts, the authorities concerned are empowered to pay Public Analysts such remuneration as shall be mutually agreed upon. Additional payments of the kind suggested can be granted, therefore, without reference to the Ministry of Health.

"We would ask you kindly to bring this letter to the notice of your authorities as soon as possible, and we shall be greatly obliged if you will inform the Registrar of the Institute (30, Russell Square, London, W.C.1) as to the emoluments attaching to the analytical appointments under your authority, and with regard to any modifications which your Council may have made recently, or may contemplate making, in the terms for such appointments.

"Yours faithfully,

"HERBERT JACKSON,

"President of the Institute of Chemistry.

"SAMUEL RIDEAL,

"President of the Society of Public Analysts.

"TO THE CLERKS TO THE COUNTY COUNCILS, CITY AND BOROUGH COUNCILS CONCERNED WITH THE ADMINISTRATION OF THE SALE OF FOOD AND DRUGS ACTS, AND FERTILIZERS AND FEEDING STUFFS ACTS."

The Joint Committee with the Institute of Chemistry has also further considered the question of the inclusion of the Public Analysts in any superannuation scheme. I gave at some length the views which had been arrived at in my Address last year, and now that the report of the Departmental Committee on the Superannuation of Persons employed by the Local Authorities in England and Wales has been presented to the Minister of Health, it is to be hoped that this difficult question will be definitely settled.

The Committee report that it is desirable that a uniform scheme of superannuation should be introduced applicable to persons employed by the local authorities in England and Wales. The scheme is a compulsory one for all Public Analysts not over fifty-five at the commencement of the scheme, and receiving not less than £50 per annum. The part-time Public Analyst is, therefore, definitely included in the scheme.

In assessing the contributions to be paid and the pensions to be received by

part-time officers, it is recommended that "the salary or wages should be the net sum received by the officer for his personal use exclusive of (1) any portion paid to assistants engaged by him, and (2) any travelling and other *expenses* of his office." "Occasional emoluments should be ignored." "Emoluments represented by fees paid to a professional officer on a recognised professional basis should be excluded."

On the other hand, the Committee considered that "the value of all permanent emoluments, which may be defined as any benefits, enjoyed in kind, attaching to an appointment, should be added to the salary or wages, and be definitely fixed by the employing authority on the basis of their value to the holder" (par. 46).

From the wording of the above extracts it seems doubtful if the Committee considered, or even were acquainted with, the widely varying conditions attaching to the appointments of part-time Public Analysts. In any case, these extracts raise some difficult points on which a decision must be come to by Public Analysts: Should a Public Analyst (part time) be pensioned on the basis of the actual fees or salary paid him, or should a deduction be made for his working expenses? If the latter, how is the amount of that deduction to be determined?

The provision for retirement at sixty, after forty years' service, to be optional on either side, obviously leaves the final decision as to whether the officer shall continue at work with the local Authority after the age of sixty if the parties are not agreed—*i.e.*, the officer can retire at sixty if he likes, but he cannot continue in office unless the Authority agree to let him.

In the case of officers not entitled to superannuation who have to retire, the provision for the return of their contributions with only 3 per cent. compound interest is hardly just, and certainly not liberal. A sliding scale of pension to reach the maximum after ten years' service would obviously be much fairer.

Where retirement takes place owing to the abolition of office full compensation should be granted, as was done in the case of the London Government Act, 1899.

In the case of voluntary retirement the return of contributions without any interest whatever is altogether unfair.

The exclusion of officers of fifty-five years and upwards, although justifiable from an actuarial point of view, will be a great disappointment to many, and if a bill embodying the scheme in its present form should be introduced into Parliament, steps should be taken to amend it in this connection, and also as regards the failure to recognise any claims in respect of "back service"—*i.e.*, past service at the time the scheme comes into force. The power given to the authorities to grant compensation or superannuation in such cases if they like is of very little value. It is probable that they have this power already, and in any event they would be extremely unlikely to exercise it in the case of a part-time Public Analyst.

The Joint Committee has also under consideration the question of revising and extending the A.R. list of reagents for analytical purposes published in 1915.

A Standards Committee has also been formed from representatives of our Society and that of the Institute of Chemistry, with members of the Society of Chemical Industry and the Institute of Metals, to consider the proposal of preparing and issuing standard alloys and like substances for future reference, which might be useful for arbitration purposes, or in cases of doubt or dispute.

We have delegated Mr. Huntly to represent the Society on a committee of the Imperial Mineral Resources Bureau, which has had several meetings during the year, and is engaged on the important inquiry as to how the scientific and technical information required for the development of our mineral resources can best be disseminated. The preparation of a general card index of the books and periodicals relating to the mining and metallurgical industries available in London and Newcastle is under discussion, and some pooling of abstracts of papers is being considered.

I have, during the past year, continued to represent this Society at the meetings of the Conjoint Board of Scientific Societies, and of the Federal Council for Pure and Applied Chemistry. Both of these bodies have had several important matters under consideration, amongst others, as you are aware from an informal report I brought to your notice at one of our sessional meetings on the proposal to develop the Metric and Decimal Systems in this country. This matter has been referred back, so there is at present nothing to report in connection with this proposal.

In my address last year I referred to the activity of the Department of Scientific and Industrial Research and its bearing on the scope and work of our members.

The Food Investigation Board has now published its report for the year 1918, and in it they point out that their field of work is distinct from that of the Medical Research Committee, the Development Commission, and of the Public Health Department of the Local Government Board, under which those of our members who hold Public Analyst appointments formerly worked.

At the same time, in the first year of work they appointed committees at the request of the Ministry of Food on matters which ultimately concern us in our official duties, such as a survey of the sources of the supply of edible oils and fats, with the object of increasing the proportion available for human consumption, and a sub-committee is at work on the problem of the evaluation of a definite chemical standard of quality which, if it can be evaluated, would be of great use in our work, and in addition, the Ministry of Food have referred to the special questions of the distribution of accessory foodstuffs (vitamines) in edible fats of various origin and the presence of traces of nickel in hardened fats. I gather that the Medical Research Committee is also engaged on research on accessory foodstuffs, and that the two committees are working in accord.

The terms of reference of the Oils and Fats Committee require them to consider and report upon :

1. The treatment of inedible oils and fats necessary to fit them for human consumption.
2. The production of glycerol from some sources other than naturally occurring esters.
3. The provision of suitable substitutes for animal and vegetable oils and fats now used in commerce.
4. The purification and digestibility of fatty acids.
5. The effect of storage and methods of treatment of fats on the fat-soluble accessory foodstuffs, and an edibility test conference has been set up to formulate a set of standards for the more common edible oils and fats, including (a) colour, (b) test of free acidity, and (c) tests for oxidation and rancidity; but excluding the question of "bouquet."

Meantime war emergency can be pleaded for the definitions of edible and inedible fat issued by the Food Controller under the order of June 12, 1918, but it would have been difficult to prove that more than 1 per cent. of water and impurities, or more than 2 per cent. of free fatty acids, ceased to make a bone fat or dripping edible, and even last year on May 30, 1919, the Food Controller seems to have taken the power to say what an oil or fat compound (other than a margarine or vegetable butter) may consist of without giving us any indication how their suitability for human consumption shall be determined; the only practicable tests under the order being now that it shall contain no other commodity (whatever that may mean) and not more than 0.5 per cent. of free fatty acids and 0.5 per cent. of water and impurities taken together. Under the same order *margarine* must now contain only such ingredients as the Food Controller may prescribe, and at least 80 per cent. of oil and fat. It might be desirable in this connection for the Ministry of Health to ask the Food Controller to prescribe the presence of the fat-soluble accessory food factor.

Vegetable Butter is now 75 per cent. of refined, deodorised vegetable oil and 25 per cent. of nut cream and no other ingredients, and yet, up to the time of sale, it may contain up to and no more than 2.5 per cent. of water.

Shredded Suet is now defined as (1) at least 80 per cent. of fat, premier jus or premier jus and beef stearine; (2) not more than 2.5 per cent. of water, and the balance hard grain rice flour, and no other commodity.

As a matter of fact I was not officially informed of this order, but by the end of October, by inquiring of the Director of Oils and Fats Branch of the Ministry of Food, I obtained a copy.

The Ministry of Health, which had been in existence for several months, could not give me any information on the subject, but informed me that there was no legislation so far as their Department was concerned, so that we may take it that the Ministry of Health is not at present engaged on the subject. It is surprising that the Ministry of Food issued these orders whilst they were themselves inquiring from the Food Investigation Committee of the Privy Council for information as to the wisdom or scientific justification of such legislation.

We thus see that certain subjects which our own Analytical Investigation Scheme was founded to cover are being dealt with under the ægis of the Privy Council, and we hope that these reports will give the Ministry of Health the information which will enable us to exercise in our official work a greater and more effective control of the food of the people.

There is, however, plenty of work for our scheme, several interesting researches are now being prosecuted by our members, and, as I pointed out last year, some fifty original investigations initiated by your Committee have already been published in the ANALYST.

One communication under this scheme is being made to the Society to-night by Mr. Trickett, and we hope to receive others at an early date.

In December last, a number of our members had an informal discussion with the members of the Agricultural Education Association with regard to the different methods of estimating crude fibre in feeding-stuffs, and from what passed it would

appear that there is room for further research in order to attain a greater uniformity in the results obtained by different methods. A proposal has been made that this matter should be investigated under the Analytical Investigation Scheme* (ANALYST, 1914, 39, 337-340), and a subcommittee has been appointed to decide upon the line the inquiry should take. The Hon. Secretaries will be pleased to receive further suggestions as to subjects for research.

By far the most important event in the year has, however, been the establishment of the new Ministry of Health. In the negotiations before this new Government Department came into being, Sir Herbert Jackson and myself, as your President, addressed a letter in March last to Dr. Addison, which was published in Part III. of the Proceedings of the Institute of Chemistry in July last, urging adequate representation of our profession on the Advisory Committees, and eventually the Institute was asked to nominate two representative chemists. Unfortunately, the Ministry have retreated from their original invitation, and now only one chemist, Sir William Tilden, F.R.S., is in charge of the various interests which Public Analysts have now and in the future to carry out.

Dr. F. Gowland Hopkins, F.R.S., and Dr. T. Eustace Hill, O.B.E., are also, however, members of the Council on Medical and Allied Services, and will be interested in our branch of public health work.

A deputation from the Association of Public Analysts of Scotland urging similar claims in connection with the Scottish Board of Health Bill was received by Mr. Robert Munroe, K.C., M.P., the Secretary for Scotland, on May 3, and an account of our meeting, which I, as your President, and Mr. P. A. Ellis Richards attended, is also recorded in the Proceedings of the Institute.

As a result of this effort, up to the present time all that has happened is this: The Association of Public Analysts of Scotland were asked by the Scottish Board of Health to nominate two members for the Medical and Allied Services Committee which is to advise the Board. As the result of that nomination, the Scottish Board of Health wrote to Dr. Tocher and asked whether he would be willing to join the Committee, and thus the profession has now only one nominee there as in England. I am sure that if he gets the opportunity he will raise the question of amending the Sale of Food and Drugs Acts, and will also press for the appointment of an Official Chemical Adviser, or expert, on the Scottish Board of Health.

June, 1919, brought about the termination of the Local Government Board as a Department of State and the consequent transference of our duties as Public Analysts appointed under that Board to the new Ministry of Health.

The routine work of the Inspectors of Foods under that Board on such matters as the administration of Unsound Food and Foreign Meat Regulations, Sale of Food and Drugs Acts, and other measures dealing with food, is now transferred to the new authority. The last report deals with a large number of special questions arising out of the abnormal conditions of food supply. Especially with regard to imported frozen meat, inquiries were held at several provincial towns and complaints in-

* A reprint of the report of the Analytical Investigation Scheme may be obtained on application to the Hon. Secretaries of the Society.

vestigated, and found to be chiefly confined to matters arising out of unsuitable methods of conveying meat by rail.

Dr. McFadden also draws attention to the liability of certain articles used in the preparation of foods to become contaminated with arsenic, but reports that no recurrence of the circumstances of two years ago have been noted. He still considers that baking powder materials are liable to suspicion, and thinks it desirable that Public Analysts throughout the country should continue to keep food materials of this class under observation locally. The antimony found in self-raising flour at Manchester was attributed to accidental bulk contamination of a limited amount of flour or of the ingredients used.

Imported tinned fruits arrived into this country under suspicion of having been maliciously infected with pathogenic bacteria, but these allegations were found to be false.

Christmas puddings and tinned meat intended for the troops were also under similar suspicion, the former bearing no evidence of unwholesomeness either culturally or as the result of feeding experiments on monkeys; but some of the tins of meat were blown.

The Government Chemist, in his Annual Report (see p. 113), states that 97 samples were referred by magistrates as against 102 in the previous year. The results of these differed from those of the Public Analyst in only twelve cases. Eighty-two of the referred cases were samples of milk.

The importance of proper inspection of home-killed meat is somewhat out of the scope of our duties, but the Public Analyst might be required to issue a certificate as to whether meat derived from a diseased animal or in an unwholesome state is of the nature, quality, and substance demanded.

In 1898 the Royal Commission on Tuberculosis recommended the closure of private slaughter-houses, but up to the present only about 100 local authorities have public abattoirs; and during the war the Ministry of Food have, under an order of that Department, made themselves responsible for the grading of cattle and their subsequent slaughter in licensed houses, and the late Local Government Board arranged for the inspection of all fourth-grade cattle killed at those slaughter-houses by the Medical Officer of Health of each district concerned, but the position in regard to this matter from the Public Analyst's point of view is most unsatisfactory. Supervision of places where food is prepared or kept for sale for human consumption was developed as a war necessity by the power given for this supervision in the contracts entered into between the Government Departments and the various food contractors, and in this way factories and other places where food is prepared or kept for sale, if carrying out a Government contract, were under supervision as to hygienic surroundings and suitability and cleanliness. This supervision will no longer obtain when Government contracts are at an end.

I hope that the improvements brought about in this way by the war may be maintained even without further powers than those already possessed by the local authorities under the Public Health Acts. Early in the year we had a discussion and two interesting papers on the "Analysis of Sausages, Meat Pastes, and Army

Rations," which show how difficult it is to ascertain whether these are made of pure and wholesome foodstuffs.

There is a growing demand for cooked and preserved foods of all kinds, especially in the poorer and in manufacturing districts. The coal shortage and high prices indicate that the number of premises where food is prepared or cooked for sale will increase, and in the absence of any fresh regulations for this class of premises, the examination and control of cooked foods under the Sale of Food and Drugs Acts becomes more and more important; whilst the difficulties of definition and the routine examination for bacterial contamination by flies, the presence of the droppings of rodents and the like, and ptomaine poisoning, renders the work of the analyst more exacting.

The Food (War) Committee of the Royal Society has brought vividly before the authorities several problems on human nutrition, which bear forcibly upon the value of food analysis in determining the nation's dietary. The energy value of foods, the relative value of the proteins, carbohydrates, and fats, and their digestibility in different forms, have all an important bearing on the question; but it is doubtful whether our knowledge of these factors is yet sufficiently advanced for these to be included in standards to be complied with which can be controlled under the Acts.

The vitamins are known to play an essential part in nutrition, yet we have no ready means of determining whether a given sample is deficient or not in these all-important constituents.

I have already mentioned that the Food Investigation Board has undertaken to elucidate this matter with regard to edible fats, although the Medical Research Committee, in their special report, No. 38, have shown that fats, like butter and cod-liver oil, contain the fat-soluble A vitamin, which insures normal growth, and the absence of rickets and defective teeth in children, but that it is absent from hydrogenated vegetable oils, and, consequently, a margarine containing no animal fat is probably deficient in food value.

The embryo of cereals and pulses also contains an antineuritic vitamin, which is water-soluble, and is absent from the white flour of the new peace bread, but was present in the war bread of 80 per cent. milling and in wholemeal.

The polishing away of the bran and embryo of rice removes its vitamin, and is known to have accentuated the spread of beri-beri in the East, and dangers of a like kind may be lurking in many of the modern methods of preserving food.

Last year I pointed out that your officials had drawn the attention of the Ministry of Food to the impossibility of insuring a scientific control of the orders which were then being issued in regard to bread and flour; but now that there is a definite Government policy of subsidising the bread of the people by £50,000,000 per annum, it should be possible at any rate to limit the quantity of water which a loaf of bread should contain. There is at least 5 per cent. variation in the amount of water from different bakeries, and seeing that an adulteration with water of milk to this extent now usually insures a heavy penalty on conviction, I do think that in this, the most important of our foods, we should at least expect to get food of standard quality and purity.

A loaf of bread is sold by weight; 5 per cent. additional water in a loaf would enable a given supply of wheat to supply additional loaves to last the country another fortnight, but would not increase its food value. This session it is proposed to legislate the sale of gas by heat units; is it too much to ask that the controlled price of the subsidised bread should be based upon its calories or food value?

The experiments carried out by the food (war) committee of the Royal Society on the digestibility of breads give us definite information on subjects which, but for the war, would probably have been left alone.

The experiments were undertaken for the purpose of showing whether the more economical milling of flour (from a 70 per cent. to a 90 per cent. basis) would have any deleterious effect on the health of the population and how it would be received by the people should the necessity arise for supplying it.

The experiments showed that in two mixed foods containing 50 per cent. of their calories in bread, the one made up with the 90 per cent. flour contained 93.4 per cent. utilisable energy and 87.4 per cent. utilisable nitrogen, while that made up from the 80 per cent. flour, 96.14 per cent. energy, and 89.4 per cent. nitrogen.

Further experiments showed that individuals fed on the 90 per cent. flour retained on the average 0.71 grm. nitrogen per day, while those fed on the 80 per cent. flour, 1.96 grms. All individuals expressed a liking for the 90 per cent. bread.

Previous experiments have shown that the digestibility of husk is increased if it be ground to a fine state of division. In these experiments the fine husk was added to the flour and the bran was quantitatively recovered by sieving with No. 7 silk (80 meshes per linear inch), and the separation formed a check on the milling. Evidently the process can be used to identify various grades of flour, and there can be no doubt that fineness of division is an important factor which might easily be standardised and thus be controllable under the Acts.

Experiments with flour containing 20 per cent. maize and ordinary wheaten flour revealed the fact that the percentage of available energy and available nitrogen was the same for both, but that the bread made from maize flour was at first difficult to swallow and was the cause of various symptoms which disappeared after a few days.

Further work on vitamins has shown that they are destroyed when fresh foods are submitted to such processes as desiccation or concentration even under reduced pressure. Simple hydrogenation of vegetable oils produces a similar result.

The antiscorbutic vitamin of fresh vegetables is destroyed by drying or by prolonged cooking so that vegetables when desiccated or tinned even in the absence of preservatives have not the food equivalent of the fresh article.

Unfortunately no routine methods of analysis have yet been devised to test their absence in foods, although from time to time methods have been advanced with that object in view. Thus Thompson (*J. Agric. Research*, 1915, 3, 425-429) found that phosphoric acid was present in organic combination with inositol or phytic acid in the seeds of many plants, and that in the case of rice the total phosphorus in the bran was 2.29 per cent., in polished rice 0.14, and in unpolished rice 0.32. Phytin itself determined by extraction with 0.2 per cent. HCl, and after precipitation with alcohol was present to the extent of 8.2 per cent. in bran but could not be obtained from

polished rice. The necessity for the proper examination of rice for facing materials is shown by the recorded case of a calculus which was obtained from a person very fond of rice and which had a composition very similar to the steatite found on the rice which he had been accustomed to consume. (ANALYST, 1910, 35, 479.)

With reference to the proposal of a standard for cheese I am sorry to say that so far no progress has been made. Irish makers who turned out 14,000 tons of cheese last year are unanimous in asking for a standard of 45 per cent. of fat in the dry matter for whole-milk cheese, and 35 per cent. of fat in the partially skimmed cheese. The Irish Department of Agriculture endorse these figures. On the other hand, the Scottish Board of Agriculture would like to see a standard of 50 per cent. of fat in the dry matter for whole milk cheese, while the English Ministry of Agriculture consider that their makers must be educated up before any action could be taken. The question has been somewhat complicated by the desire to set up standards for condensed milk. Some conferences were held with the trade on this latter subject and the following standards were agreed to :

Condensed Whole Milk.—Total milk solids not less than 30 per cent., made up as follows : Solids-not-fat, 21 per cent. ; fat, 9 per cent. ; added sugar not to exceed 42½ per cent.

Condensed Separated Milk.—Total milk solids not less than 26 per cent. ; sugar not to exceed 46 per cent.

It is found, however, that the legal advisers are of the opinion that under Section 4 of the Sale of Food and Drugs Acts, 1899, the Ministry of Agriculture had no power to set up standards for condensed milk, and the matter has been referred to the Ministry of Health for action. I wonder for how long?

I think if our Society were to lend its support to the demand for standards for all milk products, and to press this demand on the three Boards of Agriculture for England, Scotland, and Ireland, and also the Ministry of Health, something might be done.

At present the honest trader is at the mercy of the dishonest trader when marketing his cheese or condensed milk. Unlike the old question of butter and milk standards, the demand for standards is being pressed forward by the manufacturers, and although in many cases the fixing of standards has resulted in deterioration or degeneration, on the whole they work for uniformity and minimise disputes.

I have recently, in my own practice, had occasion to form an opinion on milk powder. This, as you know, forms an ingredient in proprietary foods, but whether a solid milk powder has chemical properties as to nature, quality, and substance of the milk from which it is derived, I find no binding authoritative statement.

I submit that Dr. Coult's suggested limits for the quantity of fat in full-cream and half-cream milk powders might be made known to infant welfare centres and children's homes, and that the local authorities should be pressed by the Ministry of Health to ascertain how far, in these days of dear milk, these milk powders are being used, and what is the nature and quality of the supply.

Drug samples are seldom submitted by local authorities to their analysts. Tabulated returns for the last few years are not available. In my own case I find that out of 5,619 samples examined during the last fourteen years only one drug was included.

I understand that in Lancashire, under the instructions of the Ministry of Health, drugs used for National Health Insurance dispensing are being examined under the Acts as follows :

The Medical Officer of Health, with the analyst, writes a set of prescriptions, hands them to the clerk to the Insurance Committee, and he asks doctors (five or six) from different localities to order them. These are brought in by a boy, saying he would call later on. The boy returns, and when told the medicine is ready, walks out and informs the inspector, who then comes in. The inspector divides the sample in the usual manner after paying for the medicine. Then he hands over a copy, requesting the druggist to stamp it. He keeps the copy and initials the original, which he hands back to the druggist. In about ten days postcards are sent out by the analyst that the sample need no longer be kept (this is done at the request of the Pharmaceutical Committee), while in some cases the medical officer writes that the medicine is deficient by so much per cent., and warns the druggist that prosecution will follow if deficient on another occasion.

The Medical Research Committee has also reported to the Ministry of Health on the subject of testing venereal drugs, and the following are the suggested standards for salvarsan and neosalvarsan :

SALVARSAN.

Physical Properties.—The drug should be in the condition of a pale yellow to yellow amorphous powder, freely mobile in contact with glass surfaces, and practically without odour, except that due to traces of ether.

Ash.—On ignition the yield of ash should not be more than 0·5 per cent.

Solubility.—If 0·5 grm. of the powder is added to 35 c.c. of distilled water contained in a glass-stoppered 250 c.c. cylinder and the contents vigorously shaken, the powder should rapidly and completely dissolve. The solution should be neutral to Congo red paper, and should be of a pale yellow colour, perfectly clear and free from flocculi, hairs, dust, or suspended particles of any kind. The addition of 1 c.c. of 15 per cent. sodium hydroxide to the aqueous solution should cause a preliminary separation of the solid arseno-base which, on shaking the liquid, should completely dissolve, forming a bright yellow solution. This, on dilution to 250 c.c. with 0·5 per cent. saline solution, should give a perfectly clear bright light yellow liquid.

Percentage of Arsenic.—The powder, as taken directly from the tube, should not contain less than 30 per cent. nor more than 34 per cent. of arsenic, as determined by some absolute method.

NEOSALVARSAN.

General Properties.—Neosalvarsan should be a yellow to orange-yellow fairly dense dry powder, mobile in contact with glass surfaces, and practically without odour except that due to traces of alcohol and ether ; its aqueous solution (5 per cent.) should be neutral to litmus paper.

Solubility.—If 0·6 grm. of neosalvarsan be added to 1 c.c. of water, it should dissolve rapidly and completely, forming a yellow-coloured solution, mobile, non-gelatinous, and free from suspended particles.

Percentage of Arsenic.—Neosalvarsan should contain not less than 18 per cent., and not more than 21 per cent., of arsenic. The methods of estimating the arsenic—the oxidation titration method and the gravimetric assay as magnesium pyroarsenate—are given in an appendix to the report. Biological tests are also dealt with, and it is pointed out that fresh legislation should be obtained making the Ministry of Health responsible for controlling the conditions under which such toxic drugs are supplied, and that the department should make regulations prohibiting the import and sale of the drugs unless they comply with certain stipulated chemical and biological tests.

I think that it is in one way unfortunate that so many departments are now interested in these questions, as it makes it difficult for the Public Analyst to follow the various orders and regulations which are issued, and still more at a loss to deal with what does or does not constitute adulteration or what is or is not to be regarded as injurious to health. For many years we asked for a Committee of Reference, and in our recent memorials in connection with the Ministry of Health the most urgent matter for consideration has been the want of instruction or guidance shown by the several departments in the matter of standards of purity which could assist us in our work.

Passing from a consideration of food and drugs, I am glad to be able to record that one of the first acts of the Ministry of Health has been to appoint a chemical and bacteriological inspector in the person of Dr. Calvert, for many years with Dr. Maclean Wilson of the West Riding Rivers Board. This new inspector will assist the Ministry of Health in connection with the duties involved, under the Rivers Pollution Acts, and in inquiries in relation to sewage disposal cases. Let us hope that the appointment indicates that the Ministry of Health will follow up the work of the Royal Commission on Sewage Disposal which was closed down by the Local Government Board owing to war conditions, but which has paved the way for the promotion of economical methods of sewage disposal and the prevention of the present national nitrogenous waste which obtains in most of the schemes sanctioned in the past.

I must apologise for the length of my address. I thank the officers for their assistance during my term of office and the Society for electing me to this chair, and I now have pleasure in welcoming your new President, Mr. Alfred Smetham.



AN INVESTIGATION INTO THE COMPOSITION OF THE UNSATURATED HYDROCARBONS PRESENT IN COAL-GAS.

By F. S. SINNATT, M.B.E., M.Sc.(TECH.), F.I.C., AND L. SLATER,
M.Sc. (TECH.), A.I.C.

(Read at the Meeting, February 4, 1920.)

COMPARATIVELY little information appears in the literature dealing with the composition of the unsaturated hydrocarbons occurring in ordinary coal-gas. No very recent analyses are available, and it may therefore be of some interest to place on record the results of an investigation carried out in June and July, 1914. Military duty has prevented the completion of the original investigation, but it is felt that the facts may be of some use to chemists dealing with the analysis of coal-gas, especially those who calculate the calorific value of the gas from the results obtained by chemical analysis. It is a common opinion that the calorific value of gas obtained by calculation from the analytical figures is probably quite as accurate as that obtained by the determination in a calorimeter. One of the chief sources of error in the calculated value lies in the fact that the exact composition of the unsaturated hydrocarbons is not known, and consequently the calorific value of these constituents is generally computed. The values given in this paper show that over two periods of about three weeks the composition of the unsaturated hydrocarbons present in a normal town's gas (Manchester) was practically constant, and it may be assumed, with a certain amount of reason, that coal-gas from other sources would yield very similar results.

In order to be able to elaborate a method for the extraction of the unsaturated hydrocarbons from the gas, it was necessary to ascertain whether bromine would absorb the hydrocarbons if the coal-gas were simply aspirated through it. In gas analysis it is usual to treat the gas a number of times with the reagent, in order to bring about complete absorption. Coal-gas was aspirated, at the rate of 10 litres per hour, through a series of three washing bottles, each containing bromine covered with water; the depth of the bromine was about $\frac{3}{4}$ inch, and the water about 1 inch. The first two wash-bottles were cooled in water, the last ice-cooled. When 50 litres of gas had passed, the liquid in each bottle was treated with dilute caustic soda solution to remove the excess of bromine, and the bromine compounds of the unsaturated hydrocarbons were separated. It was found that the third washing-bottle contained no liquid bromine compounds at all, whilst the second bottle contained only a small volume, indicating that the unsaturated hydrocarbons were practically completely absorbed by the bromine in the first bottle. Three washing-bottles were therefore connected to a meter, and coal-gas aspirated first through the meter, care being taken to insure the liquid in the meter was saturated with the coal-gas before commencing the actual determination. It was necessary to attach an aspirating pump to the outlet of the last washing-bottle, to overcome the pressure of the liquid in the washing-bottles. The rate at which the gas was aspirated through the bromine was about 10 litres per hour, and the experiments ran continuously from 9 a.m. to 5 p.m. for ten days in the first test, and three weeks in the second test.

The rotation of the wash-bottles was changed when the first one had become practically saturated, and fresh bromine was placed in the last. By this means, the complete absorption of the unsaturated hydrocarbons was assured. The bromine from the first wash-bottle was collected in one vessel, until the experiment was complete, and was then treated with a very dilute solution of caustic soda; it was washed repeatedly with small volumes of water, and the colourless bromine compounds of the unsaturated hydrocarbons were allowed to stand over anhydrous potassium carbonate until completely dry. The liquid was finally filtered through dry filter-paper. Throughout the above operations the temperature of the liquid was kept as low as possible in order to prevent excessive evaporation.

The liquid was subjected to distillation under a pressure of 15 mm., and the distillates collected at the following temperatures: up to 100° C., and from 100° C. to a point where decomposition occurred (145° to 150° C.). The distillate collected below 100° C. (15 mm.) was distilled under a fractionation column, and yielded the following results:

			First Experiment.	Second Experiment.
Total weight of liquid obtained	...	...	235 grms.	520 grms.
<i>Fraction °C.—</i>				
Up to 125	...	...	4.4	17.1
125 to 135	...	...	141.7	280.5
135 to 145	...	...	24.4	42.3
145 to 165	...	...	3.4	9.0
165 to 195	...	...	2.0	6.3
Residues from both distillations not identified, but containing dibrombenzene	...	...	59.1	164.8
Volume of gas	...	...	700 litres.	1,600 litres.

The residue remaining from the distillation under reduced pressure, and the redistillation under ordinary pressure, was not investigated, beyond proving that the chief constituent was dibrombenzene (m.p. 87° C.).

Fraction °C.	Chief Constituent.
Up to 125	A mixture of benzene and toluene, and a small percentage of heptane.
125 to 135	Ethylene dibromide, b.p. 130.3° C.
135 to 145	Propylene dibromide, b.p. 141.5° C.
145 to 165	Butylene dibromides, b.p. 148° to 166° C. (brombenzene could not be identified).
165 to 195	Amylene bromides, b.p. 172° to 178° C.

The whole of the above compounds, and also the hexylene bromides, boil practically without decomposition below 100° C. under a pressure of 15 mm.

Acetylene tetrabromide, which is formed by the action of bromine upon acetylene,

has a boiling-point of 114° C. at 12 mm. pressure, and could not therefore be present in the distillates obtained during these experiments, where the limiting figure was 100° C. at 15 mm. pressure. The dibromide of acetylene is not formed by the action of concentrated bromine upon acetylene. It is clear, therefore, that the only compounds which have been separated by the treatment described in this paper are the members of the ethylene series.

The percentage composition by volume of the unsaturated hydrocarbons (to the first decimal) present in the gas, calculated from the weight of dibrom compounds obtained, is as follows :

	First Experiment. Percentage by Volume.	Second Experiment. Percentage by Volume.
Ethylene	83.8	84.3
Propylene	13.4	11.8
Butylene	1.7	2.3
Amylene	1.0	1.5

Repeated determinations of the actual percentage of unsaturated hydrocarbons in the coal-gas were carried out during the progress of the experiments, using fuming sulphuric acid as the absorbent, and the amount varied between the limits of 3.5 and 4.5 per cent. It is evident from the values found, that although the actual composition of the unsaturated hydrocarbons showed comparatively little alteration, the proportion of benzene, toluene, etc., to unsaturated hydrocarbons in the gas, must have varied considerably during the course of the experiments. If other experimentors wish to carry out similar determinations, it may be suggested that it would be advisable to eliminate the aromatic hydrocarbons, before attempting to absorb the unsaturated hydrocarbons in bromine. The fraction from 100° to 140° C. (15 mm.) became solid on being allowed to stand. It was not completely investigated beyond finding that its melting-point was 87° C., and that the main compound present was dibrombenzene.

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DISCUSSION.

Mr. CHASTON CHAPMAN remarked that he was rather surprised that it had been found possible to estimate by a method depending on the separation by fractional distillation under reduced pressure of their mixed bromides, four adjacent homologues such as ethylene, propylene, butylene and amylene, with the degree of accuracy suggested by the figures the authors had given. The boiling-points of the bromides in question were not very far removed from one another, and apart from any interference due to the presence of unsaturated hydrocarbons belonging to other series, he would scarcely have expected that such a method would have been capable of giving quantitative results.

Mr. H. G. COLMAN said that the method adopted by the authors—namely,

extracting the unsaturated hydrocarbons by bromine and fractionation of the resulting bromides—did not seem likely to give any definite information with respect to the unsaturated hydrocarbons present in only small quantity. Undoubtedly the nature and relative amounts of the various unsaturated hydrocarbons varied very greatly according to the temperature and other conditions under which the coal was carbonised, and still more in the case of gas made from oil. In all cases olefines formed the largest proportion of such hydrocarbons, but varying amounts of dienes, acetylenes and cyclodienes were always present.

So far as the constituents having five or more atoms of carbon are concerned, the most promising material for their investigation appeared to be the crude benzol obtained by washing coal-gas with oil, which always contained these hydrocarbons in considerable quantity.



THE ESTIMATION OF THE AVAILABLE OXYGEN IN SODIUM PERBORATE AND IN PERBORATE SOAP POWDERS.

By H. TRICKETT, F.I.C.

(*Read at the Meeting, February 4, 1920.*)

Of the many methods that have been proposed for the estimation of the available oxygen in hydrogen peroxide and sodium peroxide, most are applicable to the valuation of sodium perborate, and the volumetric determination, either with potassium permanganate (1) (Jaubert, *Comptes rend.*, 1904, **139**, 796), or with potassium iodide (2) (Kingzett, *J. Chem. Soc.*, 1880, 792; Carpenter and Nicholson, *ANALYST*, 1884, **9**, 36), is generally regarded as the most accurate. Since some determinations by these methods were found to give varying results, it seemed of interest to examine the conditions necessary to obtain concordant data, especially as a basis for a method of estimating the perborate contents of soap powders and similar products.

As the initial material for these experiments a sample of sodium perborate was employed, which gave the following results upon analysis:

	Found.	Calculated for $\text{NaBO}_3 \cdot 4\text{H}_2\text{O}$
H_2O	45.42 per cent.	46.78 per cent.
Na_2O	20.61 "	20.12 "
B_2O_3	24.21 "	22.72 "
O_2	9.62 "	10.38 "
Iron and chlorine	... traces.	

The values obtained indicate that the sample did not contain the full proportion of water of crystallisation. The value for the available oxygen was the mean of a number of estimations by the potassium iodide method given below.

The following methods for the estimation of the available oxygen were examined:

1. ESTIMATION BY POTASSIUM PERMANGANATE.

The permanganate method of estimation was examined by: (a) Titrating back the excess of permanganate with oxalic acid; (b) direct titration; (c) gas volumetric estimation.

(a) In this case a known weight of the perborate was washed into an excess of $\frac{N}{10}$ potassium permanganate, acidified with concentrated sulphuric acid sufficient to give a 15 to 20 per cent. solution on the final volume. The excess of permanganate solution was titrated at about 70° C. with $\frac{N}{10}$ oxalic acid. The estimation was carried out with the solid perborate, in order to test the applicability of the method to the direct estimation of perborate in soap powders. The results were rather irregular, and out of seven determinations the available oxygen value varied between 8.26 per cent. and 9.46 per cent.

(b) In the direct titration about 20 c.c. of $\frac{N}{10}$ potassium permanganate were acidified with varying quantities of sulphuric acid, and titrated with a 0.5 per cent. perborate solution at varying temperatures. It was found that direct titration gave irregular results, owing to the end-point being rather indefinite. This difficulty was easily overcome by the addition of a slight excess of the perborate solution, and titrating back with potassium permanganate. Increase in temperature and in the concentration of sulphuric acid tend to give too high results.

The most favourable conditions indicated are—

(1) Temperature for titration of permanganate, 15° to 20° C. (2) Concentration of about 5 per cent. of sulphuric acid on the total volume.

The results obtained under these conditions were somewhat low but consistent; a series of estimations gave the following results: 9.39, 9.51, 9.45, and 9.46 per cent. available oxygen as compared with 9.62 per cent. obtained by the iodide method to be described below.

Milbauer (*J. Prakt. Chem.*, 1918, **98**, 1-8) recommends the introduction of boric acid to permanganate titrations of hydrogen peroxide in order to prevent the catalytic decomposition of the latter by the MnSO_4 produced in the reaction. Although boric acid was present in these solutions, it was probably not in sufficient quantity to prevent some loss of oxygen in the method.

(c) The gas volumetric method for the valuation of perborate by means of acidified permanganate has been proposed by Bosshard and Zwick (Zeitsch. angew. Chem., 1910, **23**, 1153-1154; ANALYST, 1910, **35**, 372) and others. The results obtained were, however, very irregular, and showed variations of from 1.5 to 2 per cent. in the available oxygen. The method is rapid, but too unreliable even for approximate determinations. Cf. Archbutt (ANALYST, 1895, **20**, 4).

2. ESTIMATION BY MEANS OF POTASSIUM IODIDE.

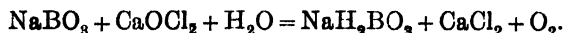
In this method, as is well known, the perborate solution is added to acidified potassium iodide solution, and the liberated iodine titrated with sodium thiosulphate, using starch as an indicator. Unless sufficient free hydrochloric acid is present, the decomposition proceeds very slowly. An acidity of 10 per cent. hydrochloric acid on the final volume gave the best results, and allowed of the completion of the titration

in twenty minutes. It was also found advantageous to proportion the concentration of the potassium iodide and sodium perborate solutions at 40:1, and to adjust the volume of the potassium iodide solution so as to give a final concentration representing about 1 per cent. of potassium iodide. The following data typically indicate the concordant results obtained: 9.65, 9.61, 9.62, and 9.60 per cent. available oxygen.

3. GAS VOLUMETRIC ESTIMATION BY HYPOCHLORITE.

Although the iodide method as described above is quite reliable, it is less rapid than a gas volumetric method, and in the case of soap powders and the like, the previous removal of the soap, etc., is necessary.

It was from this standpoint that experiments were carried out to ascertain whether satisfactory results could be obtained by decomposition of a perborate with hypochlorite on the lines of the corresponding reaction with hydrogen peroxide:



The method adopted was as follows: About 0.2 grm. of sodium perborate was mixed with about 20 c.c. of water in a beaker, the coarser lumps crushed, and the whole thoroughly wetted. The mixture was then transferred to the outer vessel of a nitrometer decomposition bottle, and the inner vessel filled with a clear saturated solution of bleaching powder—about 15 c.c. The determination was carried out with the usual precautions, and the volume of oxygen measured after five minutes' intermittent shaking of the reaction mixture. The following results were obtained on the original sample, which had been stored for a long time: 8.43, 8.32, 8.34, and 8.54 per cent. available oxygen, giving a mean of 8.40 per cent., which compares favourably with the value of 8.45 per cent. as found by the iodimetric method. This method was found equally applicable to soap powders, and could be applied directly to such powders without any preliminary separation of soap or fatty acids or decomposition of the sodium carbonate usually present in such mixtures.

To test the method in this connection, a mixture was made up of 90 per cent. soap-powder and 10 per cent. of a sample of perborate having an available oxygen value of 8.45 per cent., both having been previously graded through a 40-mesh sieve. The following results were obtained: 0.86, 0.86, and 0.84 per cent. available oxygen, corresponding to 10.1, 10.1, and 10 per cent. sodium perborate.

As a further comparison with the iodide method, a mixture was made up containing approximately 13 per cent. sodium perborate at 8.45 per cent. available oxygen, 12 per cent. borax, 25 per cent. sodium carbonate, and 50 per cent. soap powder, from which the fatty acids were removed with ignited kieselguhr according to Litterschied and Guggiari's method (*Chem. Zeit.*, 1913, 37, 677, 690). The perborate in the resulting filtrate was titrated by the iodide method, and gave a value of 1.24 per cent. available oxygen. The direct estimation by hypochlorite gave 1.13 per cent. available oxygen.

From this comparison the accuracy of the gas volumetric method with hypochlorite is satisfactorily confirmed. It should accordingly prove of value not only

on account of the advantages referred to, but also because it avoids the use of an acid solution, so that no inconvenience is caused in the estimation by the presence of carbonates in soap powder preparations.

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THE DIFFERENTIATION BETWEEN POOR AND ADULTERATED MILK.

By T. R. HODGSON, M.A., F.I.C.

LYTHGOE (*J. Ind. and Eng. Chem.*, 1914, **6**, 899), as the result of the examination of 600 to 700 samples of milk in the laboratory of the Massachusetts State Board of Health, proposes to distinguish between poor and adulterated milk by calculation of the percentage of milk sugar present in the sample, from the percentage of fat and the percentage of total solids. Starting with Olson's formula (*J. Ind. and Eng. Chem.*, 1909, **1**, 256) for the calculation of proteids from the total solids, $P = TS - TS/1.34$; and with Van Slyke's formula (*J. Amer. Chem. Soc.*, 1908, **30**, 1166) for the calculation of the proteins from the fat, $P = 0.4(F - 3) + 2.8$, he has evolved the two following formulæ for the calculation of the milk sugar :

$$(a) S = TS - [F + 0.7 + (TS - TS/1.34)]$$

$$(b) S = TS - [F + 0.7 + \{0.4(F - 3)\} + 2.8]$$

and it is suggested that "the value of S obtained by both formulæ is nearly the same when the milk is pure, and varies from 4.5 to 5 per cent.; in skimmed or watered milks the values disagree, and are above 5 in the former and below 4 in the latter."

If this fact can be established, the Public Analyst will at last be in a position to rebut easily that familiar and ever-recurring defence, "the sample was sold exactly as it came from the cow." With a view to testing the statement, 100 samples, received for analysis under the Sale of Food and Drugs Acts and reported upon as genuine, were chosen at random from a large number of samples, and the values of S for both formulæ were calculated.

The greatest difference between S (a) and S (b) was 0.61.

The smallest difference between S (a) and S (b) was 0.01.

Percentage of samples showing a difference less than 0.1	...	...	39
"	"	between 0.1 and 0.15	25
"	"	" 0.16 and 0.20	21
"	"	" 0.21 and 0.25	6
"	"	" 0.26 and 0.30	5
"	"	" 0.31 and 0.35	3
"	"	" 0.36 and 0.40	Nil
"	"	" 0.41 and 0.45	Nil
"	"	over 0.45	1

Number of samples giving a value for S (a) under 4.0	...	...	Nil
" " " between 4.0 and 5.0	...	...	58
" " " over 5.0	...	...	42
" " " of S (b) under 4.0	...	...	Nil
" " " between 4.0 and 5.0	...	...	39
" " " over 5.0	...	...	61

The following six samples show a value for both S (a) and S (b) above 5.0, and, therefore, on the formulæ are skimmed :

Fat.	Solids-not-Fat.	S (a).	S (b).
4.2	9.55	5.36	5.57
4.1	9.40	5.27	5.46
4.0	9.29	5.22	5.39
3.9	9.26	5.22	5.40
3.8	9.23	5.22	5.41
3.7	10.84	6.45	7.06

It is obviously possible for a genuine milk to show a large difference, as it could not be argued, with any hope of success, that any of the above samples had been adulterated. On the basis that samples giving a value above 5.0 are skimmed, and below 4.0 are watered, at least 42 per cent. of the samples which complied with the Board of Agriculture standard had been deprived of a portion of their fat.

The values of S (a) and S (b) were then calculated on 100 samples, received for analysis and chosen at random, which did not comply with the Board of Agriculture standard and were reported upon as adulterated, with the following results :

The greatest difference between S (a) and S (b) was 0.54.

The smallest difference between S (a) and S (b) was 0.01.

Percentage of samples showing a difference less than 0.1 ... 15

" " " between 0.1 and 0.15 ... 13

" " " " 0.16 and 0.20 ... 12

" " " " 0.21 and 0.25 ... 11

" " " " 0.26 and 0.30 ... 22

" " " " 0.31 and 0.35 ... 12

" " " " 0.36 and 0.40 ... 9

" " " " 0.41 and 0.45 ... 5

" " " over 0.45 ... 1

Number of samples giving a value of S (a) under 4.0 ... 5

" " " between 4.0 and 5.0 ... 32

" " " over 5.0 ... 63

" " " of S (b) under 4.0 ... 11

" " " between 4.0 and 5.0 ... 22

" " " over 5.0 ... 67

The following six samples show a value for both S (a) and S (b) falling between 4.0 and 5.0, and, therefore, according to the formulæ, are poor, but genuine samples

Fat.	Solids-not-Fat.	S (a).	S (b).
2.86	8.33	4.79	4.89
2.85	8.06	4.59	4.62
2.55	8.15	4.74	4.83
2.27	8.19	4.84	4.98
2.23	7.52	4.35	4.33
2.10	7.61	4.45	4.47

It is possible that there may be a little difficulty in persuading an English Public Analyst to realise that the above samples are "poor, but genuine." No less than 22 per cent. of the samples which failed to comply with the Board of Agriculture standard would, on these formulæ, have to be passed as genuine.

The values for S (a) and S (b) were then calculated on twenty samples of milk, which had admittedly been skimmed :

The greatest difference between S (a) and S (b) was 0.79.

The smallest difference between S (a) and S (b) was 0.03.

Percentage of samples showing a difference of less than 0.1 ... 25

" " " between 0.1 and 0.15 ... 10

" " " " 0.16 and 0.20 ... 5

" " " " 0.21 and 0.25 ... 5

" " " " 0.26 and 0.30 ... 10

" " " " 0.31 and 0.35 ... 5

" " " " 0.36 and 0.40 ... 5

" " " " 0.41 and 0.45 ... 5

" " " over 0.45 ... 30

Percentage of samples giving a value for S (a) under 4.0 ... 25

" " " between 4.0 and 5.0 ... 40

" " " over 5.0 ... 35

" " " for S (b) under 4.0 ... 30

" " " between 4.0 and 5.0 ... 25

" " " over 5.0 ... 45

The following five samples, although admittedly skimmed, show a value for both S (a) and S (b) between 4.0 and 5.0, and, therefore, according to the formulæ, are "poor, but genuine":

Fat.	Solids-not-Fat.	S (a).	S (b).
1.61	7.88	4.77	4.94
0.57	6.76	4.20	4.23
0.42	7.36	4.69	4.89
0.39	6.75	4.24	4.29
0.30	7.31	4.68	4.89

It would be ludicrous to describe any of the above samples as "poor, but genuine." No less than 25 per cent. of the samples of skimmed milk gave a value for both $S(a)$ and $S(b)$ falling between 4.0 and 5.0.

It is quite obvious that a sample which has been adulterated may give a value nearly the same for both $S(a)$ and $S(b)$, and also give a value falling between 4.0 and 5.0; the formulæ, therefore, show no advantage over the Board of Agriculture standard, especially if that standard is administered, as in due course it will be administered, under the provisions of The Milk and Dairies (Consolidation) Act, 1915.

Further, in this connection, attention may well be drawn to the now well-recognised formula, demonstrated by Richmond ("Dairy Chemistry," p. 152), that watered milk may easily be distinguished from abnormal milk by a consideration of the ratio of lactose, protein, ash. Richmond has shown that this ratio is extraordinarily exact, and that the average proportion is 13 : 9 : 2. He has determined the milk-sugar, protein, and ash on a large number of samples, and, by plotting the figures thus obtained against the average figures for solids-not-fat, has shown that well-defined breaks occur between 8.8 per cent. and 8.9 per cent. and between 8.4 per cent. and 8.5 per cent., thus demonstrating that a naturally abnormal milk may be distinguished from a watered milk by a marked departure from the ratio.

34, JOHN DALTON STREET,
MANCHESTER.

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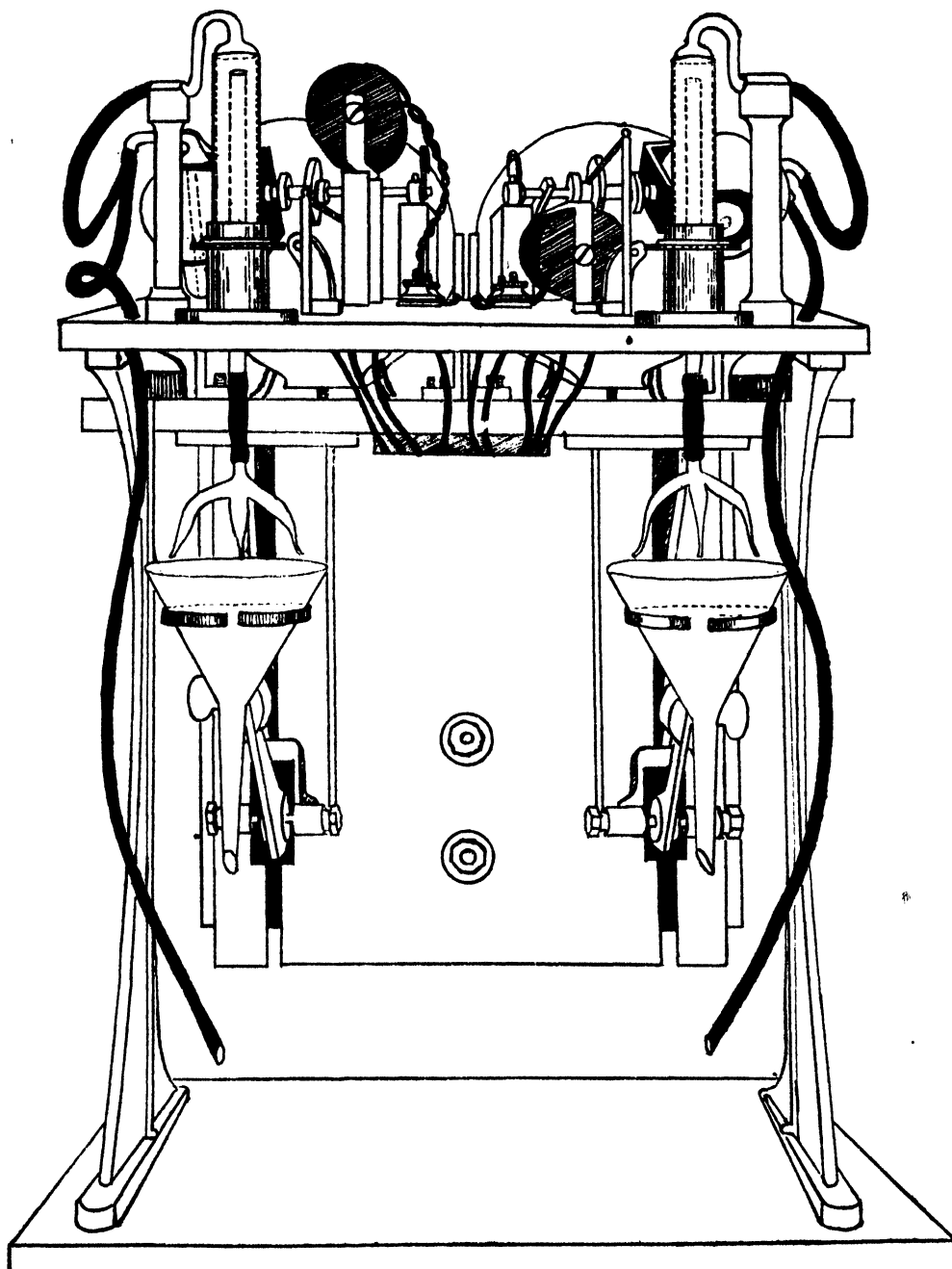
MACHINE FOR WASHING PRECIPITATES.

By ERIC SINKINSON, D.I.C., A.I.C.

THE need for automatic appliances is becoming more apparent every day in institutions where rising costs limit the scope of work, as in the chemical laboratory. The time of the analyst is largely taken up with filter-washing, decanting, and other tedious operations which must be carried out with great precision and care, yet might be applied to advantage in other directions if released from these somewhat mechanical duties.

The machine described below has been devised to wash precipitates in as expeditious a manner as possible, with hot or cold water, dilute acid or alkalis, as required, with probably the highest degree of precision possible.

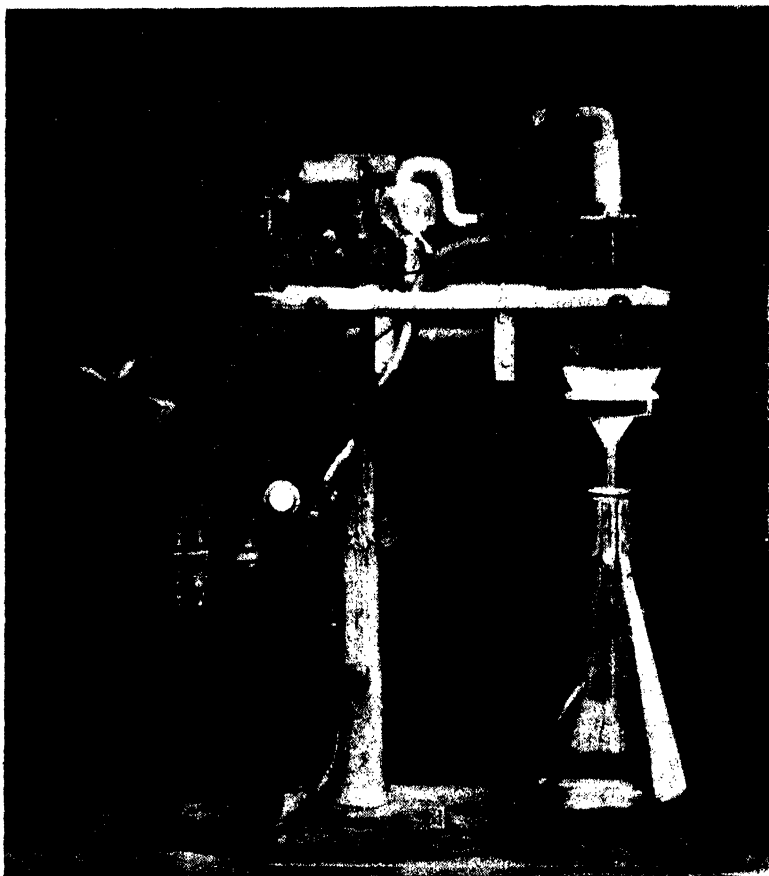
The principle employed in its construction is the rocking arm. At one end is a split ring bevelled so as to hold a filter funnel of the usual type employed in analytical laboratories; at the other is a counterpoise to adjust for different weights of funnel. Also a subsidiary arm carries a commutator to reverse a motor, according to the position of the arm, which has a free movement of a quarter of an inch between two stops. The motor drives a mercury valve by means of a worm gearing fitted to the countershaft. When the motor is run in one direction the valve closes, and on reversal opens. The valve is connected at one side to the distilled water supply, etc., and at the other to a jet which can be rotated either by a second motor, or, in



the modified form of machine, by the motor which controls the valve. The jet is connected to the valve through a sleeve partially filled with mercury, thus

permitting its rotation. The nozzle is arranged to direct the wash liquid round the edge of the filter paper in the funnel.

When working, the machine is connected up to an electric supply of suitable voltage, and the valve inlet to the water-supply, which latter may be contained in a tinned copper vessel, or glass flask, or even a thermos flask; in the two former cases it is heated by a burner, in the latter hot water may be introduced.



A precipitate contained by a filter paper in the funnel is washed by installing the funnel in the split ring at one end of the rocking arm; the counterpoise at the other end is adjusted to allow for the weight of the funnel and a suitable weight of water, which determinines the operation of the machine. The current is turned on and water flows through the valve to the jet which rotates and thus completely sprays the filter paper. When a certain weight of water has flowed in, it presses down the rocking arm holding the funnel, and this reverses the current; this, by reversing the motor, causes the valve to return to the closed position, and the water stops flowing. At the same time the jet ceases to rotate. In this way

there is economy in current because none is being used during the period of outflow from the funnel into the beaker or flask placed to receive the filtrate. As soon as the funnel becomes "light" again the valve is reversed by the lifting of the funnel arm, and water flows through the rotating jet. So the operation of washing proceeds.

If the hot washing water be contained in a thermos flask, and a glass float introduced connecting a mercury cut-out placed in the main circuit of the machine, an adjustment can be made to stop the working of the machine after a pre-determined quantity of water has passed through the filter. For, as the water flows from the flask, the float descends and causes the iron rod connected to it to break contact from the mercury in the tube, through which current is supplied to the motors of the washer. The circuit is broken and the operation of the machine stopped. By this arrangement the washer can be left entirely to itself to carry out a filter-washing and automatically to check its action on completion.

The machine as described, with only one filter-washing unit, will not meet the requirements of an ordinary silicate analysis, in which the silica, lime, magnesia and sulphur may be washed rapidly, but the alumina may take a comparatively long time. Therefore a two-unit machine was constructed for this service. In order to cheapen the constructional work, it was found that one motor could be used instead of two by small modifications of the drive, to actuate both the valve and the rotating jet without in any way complicating the mechanism. As the electric motors are the most expensive parts of the apparatus, this modification made it possible to construct a double-unit machine on one base at a cost little above that for the single unit.

The diagram gives some idea of the appearance of the double-unit machine with the working parts exposed. These are covered in normally by aluminium covers, as are the contacts of the commutator, to render them dustproof. In addition, small improvements have been introduced, such as (i.) a triple jet for more effective and quicker working, and (ii.) double-walled valve and leading tubes, the annular spaces of which are exhausted to conserve the heat of the wash-water when the machine is washing slowly.

The advantages claimed for the apparatus are :

- (1) Time saving.
- (2) Great precision in washing.
- (3) Uniformity of washing.
- (4) Washing stopped automatically if the machine is left to itself.
- (5) Simplicity of construction ; all moving parts can be adjusted by the chemist himself.

In conclusion, the author thanks Miss W. Sinkinson of the University of Sheffield for the drawing of the double unit machine used to illustrate the text.

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NOTE.

The Editor desires to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

THE ANALYST AND THE BEER PRICES AND DESCRIPTION ORDERS.

As a result of representations made by brewers, the Ministry of Food, after consultation with the Commissioners of Customs and Excise, issued in April, 1918, an instruction to all Local Food Committees. This instruction was to the effect that proceedings should not be instituted as regards the gravity of beers for which maximum prices are fixed, unless, after an allowance of 1 degree of original gravity either way, it could be proved that an offence had been committed (*Brewing Trade Review*, 1918, p. 95).

Although the analyst is concerned only with the original gravity as determined by him, yet in the course of professional practice it is well for him to be aware of this instruction (*loc. cit.*), as it is by no means uncommon for the Food Committee to rely solely on the figure returned by the analyst. It would be advisable for the analyst to point out to the Food Committee that this allowance should be made.

It may, perhaps, not be out of place to remind analysts that in cases of surcharges on worts made by Commissioners of Customs and Excise, 0.75 degree is deducted by the Principal Government Chemist from his original gravity determination before such surcharge is calculated (Finance Act, 1914, Section 7, Session 2).

EDITOR.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Estimation of Arsenic in Organic Compounds. L. J. Rogers. (*Chem. Trade J.*, 1920, **66**, 13.)—The sodium peroxide fusion of organic arsenic compounds was found to lead to a loss of arsenic, while the Johnson and Chittenden, as well as the Fresenius and Babo, methods also proved unsatisfactory. A rapid complete decomposition of organic arsenic compounds was finally achieved by employing ammonium persulphate in nitric acid solution, a method suggested by the decomposition of the dimethylglyoxime precipitate of nickel previous to cyanide titration. Half a grm. of the compound is transferred to a 500 c.c. Erlenmeyer flask, dissolved in 10 c.c. of water, and 5 c.c. nitric are then added. The whole is gently heated, and ammonium persulphate added in lumps until the solution is colourless, which usually occurs in fifteen to twenty minutes. The volume is made up to 100 c.c., and 5 c.c. of saturated microcosmic salt solution added; magnesia mixture is added in excess (about 40 c.c.), and if a precipitate forms, it is dissolved in dilute nitric acid. The whole is kept at nearly boiling-point, and excess of ammonia added in

the usual manner. The mixed precipitate of the phosphate and arsenate of ammonium and magnesium is cooled, allowed to stand about two hours, and then filtered and washed with dilute ammonia, followed by potassium iodide reduction and titration with thiosulphate. This co-precipitation was first employed by Berntrop, and effects a very perfect precipitation of the arsenic (*Zeitsch. fur anal. Chem.*, 1902, **41**, 11). The author believes the method to be superior to Lehmann's procedure, which has been adopted in some countries as the standard method for governmental inspection of salvarsan.

H. F. E. H.

Behaviour in Marsh's Apparatus of Certain Organic Arsenic Compounds used as Drugs. D. Ganassini. (*Boll. Chim. Farm.*, 1919, **58**, 385-390; through *J. Soc. Chem. Ind.*, 1920, **39**, 42A.)—The introduction of platinum chloride into the hydrogen flask of Marsh's apparatus retards or prevents the appearance of the arsenic deposit in the case of cacodylic acid. In the presence of a large excess of platinum chloride cacodylic oxide forms a yellow precipitate of cacodyl platini-chloride. If, however, only a small amount of platinum chloride is introduced, cacodylic oxide is evolved sooner or later. Even for the detection of mineral arsenic it is not advisable to use platinum chloride, since arsenic may be fixed as platinum arsenide, with a loss of as much as 50 per cent. of the arsenic present. The yellow ring, probably of erythrarsine, produced, together with the ordinary black deposit, by cacodylic acid in the Marsh test is a characteristic reaction. It may be obtained quantitatively by immersing the evolution flask of Marsh's apparatus in a bath of molten lead (about 335° C.). Atoxyl and salvarsan give the ordinary arsenic deposit in Marsh's test, but neosalvarsan and methylarsenic acid behave in an analogous manner to cacodylic acid, giving a yellow ring under the same conditions. In the case of cacodylic acid, however, the gas forms dense white fumes, and when conducted into Bettendorf's reagent (stannous chloride solution saturated with hydrogen chloride), it does not form a yellow precipitate. In the case of methylarsenic acid the formation of the yellow ring is not accompanied by the appearance of white fumes, and the gas gives an abundant precipitate with Bettendorf's reagent. The lemon-yellow ring given by neosalvarsan may be distinguished from the other two by the fact that it is only formed at a high temperature, does not change to black, and is rapidly dissolved by ammonia solution.

Application of Silicotungstic Acid to Estimation of Alkaloids. E. Taigner. (*Zeitsch. anal. Chem.*, 1919, **58**, 346-352.)—In estimating alkaloids in very small quantities of material, the author investigated the properties of the silicotungstic acid compounds of atropine, strychnine, and cocaine, said by Bertrand (*Bull. Soc. Chim.*, 1903, **21**, 434) to have the composition $12\text{WO}_3 \cdot \text{SiO}_2 \cdot 2\text{H}_2\text{O} \cdot 4$ alkaloid. This composition is confirmed for the substance dried to constant weight at temperatures not exceeding 40° C. Drying to constant weight at 40° C. is slow, but it is found that the whole of the water can be expelled at 100° C. the composition of the compounds being then $12\text{WO}_3 \cdot \text{SiO}_2 \cdot 4$ alkaloid. Drying to constant weight even at 100° C. is slow, but constant weight is quickly reached

at 120° C., whilst the compounds are not decomposed below 150° C. if nearly dry. When very moist they melt at about 100° C.

The alkaloid in presence of hydrochloric acid is precipitated by means of a 10 per cent. solution of silicotungstic acid, sodium chloride is added, and the precipitate is filtered off and washed with 1 per cent. hydrochloric acid, dried in a steam oven and then at 120° C., and weighed.

G. C. J.

Moisture Content of Cereals. O. A. Nelson and G. A. Hulett. (*J. Ind. and Eng. Chem.*, 1920, 12, 40-45.)—The method used for the estimation of moisture in cereals and other colloidal organic substances consisted essentially in heating the material under a very low pressure for definite periods of time. The water liberated was condensed in a small tube surrounded by solid carbon dioxide. The pressure maintained in the apparatus was often less than 0.0001 mm.; a Töpler mercury pump was used for the purpose, and gases, other than water vapour, evolved as the result of decomposition, were collected, and their volume measured. In this way the smallest amount of decomposition could be measured, and it could be ascertained at what temperature and for what length of time a substance could be heated without yielding an appreciable amount of water due to decomposition. When measurable decomposition occurred, observations of the evolution of gases and water gave an indication of the rate of decomposition at the temperature employed. The moisture-temperature curves obtained for wheat flour, maize flour, maize starch, cellulose (filter-paper and cotton-wool), and edestin, did not show any distinct break before a temperature of 184° C. was reached, and the heating continued for three hours or longer. All moisture had not been evolved at the time decomposition commenced, and the flat parts of the curves were extrapolated to 250° C., at which temperature it was not possible for any absorbed water to be present. The results obtained for the cereal flours were about 1 per cent. higher than those found by heating at 100° C. *in vacuo*.

W. P. S.

Purified Cresol (Cresylic Acid). H. C. Hamilton. (*J. Ind. and Eng. Chem.*, 1920, 12, 50-51.)—The following method is recommended for the purification of crude cresol. The substance is dissolved in water containing an equivalent quantity of sodium hydroxide, using sufficient water to form a 25 per cent. sodium cresylate solution; the solution is then boiled, or steam is passed through it until all odour of pyridine, etc., has been removed. Water should be added from time to time to replace that lost by evaporation. After cooling, floating oils may be skimmed off, whilst any remaining naphthalene is removed by filtration. The solution is then treated with a quantity of sulphuric acid equivalent to the sodium hydroxide used in the first place, the free cresol is separated carefully from the sodium sulphate solution (the cresol forms a supernatant layer over the latter), and distilled. Observation of the temperature at the end of the distillation permits the separation of higher boiling phenols from the cresol.

W. P. S.

Estimation of Insoluble Solids in Jams, Preserves, and Marmalades. C. A. Clemens. (*J. Ind. and Eng. Chem.*, 1920, 12, 48-49.)—Twenty-five grms.

of the sample are mixed with 100 c.c. of water, boiled for five minutes, and the liquid filtered through an alundum crucible to which suction is applied. The insoluble residue is washed with hot water, then with alcohol and ether, dried at 100° C., and weighed. The alundum crucible has many advantages over the usual linen filter used for the purpose.

W. P. S.

New Hexabromide Method for Linseed Oil. L. L. Steele and F. M. Washburn. (*J. Ind. and Eng. Chem.*, 1920, 12, 52-59.)—Various published methods for the estimation of the hexabromide yield of linseed oil have been examined and found to give results which are not concordant. The following method has been developed with the object of removing this disadvantage. One gram. of linseed fatty acids is dissolved in a centrifuge tube in 10 c.c. of dry chloroform containing 3 per cent. (by volume) of absolute alcohol, the solution is cooled to -5° C., and bromine solution (bromine 1 volume, chloroform, as above, 2 volumes) is added slowly until an excess is present. The mixture is maintained for ten minutes at -5° C., the solvent then evaporated at 50° C. under a pressure of not more than 40 mm., and the residue heated at 55° to 60° C. below 40 mm. pressure for a further fifteen minutes. The tube and its contents are now cooled in ice-water, and the residue is washed three times with ether saturated with hexabromide at 0° C.; 20 c.c. of the cold ether are used each time and separated by centrifugal action. After drying at 60° to 70° C., the tube and its contents are weighed. Results are recorded showing that the hexabromide yield obtained in this way is a more constant value than is the iodine value of linseed oil. In the case of eight samples of genuine linseed oil, having iodine values varying from 181 to 185, the hexabromide yields ranged from 45.6 to 46.9 per cent. Cottonseed oil and tung oil gave no visible amounts of hexabromide, whilst soy bean oil yielded 2.2 per cent.

W. P. S.

Effect of Mould on the Oil in Maize. F. Rabak. (*J. Ind. and Eng. Chem.*, 1920, 12, 46-48.)—To ascertain the effect of mould growth on the oil contained in maize, a quantity of the latter was placed in a large stone jar and inoculated with *Penicillium*. Samples were taken at intervals during a period of ninety days, four samples being obtained in all, and these were examined. At the end of the ninety days the maize formed a dirty grey, mouldy, solid mass, having a strong, offensive odour. The results of the examination of the oils separated from the different portions are given in the following table, the first sample representing the maize before inoculation. The yield of oil is calculated on the dry substance.

	Yield Per Cent.	Acid Value.	Saponi- fication Value.	Iodine Value.	Reichert- Meissl Value.	Soluble Acids (as Butyric), Per Cent.	Insoluble Acids, Per Cent.	Acetyl Value.	Unsaponi- fiable Matter, Per Cent.
1.	5.58	18.6	190.8	121.0	12.8	1.3	98.6	15.3	4.13
2.	4.33	46.7	191.7	121.3	2.95	1.5	90.5	10.6	9.9
3.	2.67	84.6	192.4	120.4	2.23	3.28	92.3	61.1	10.8
4.	2.06	68.7	185.1	119.0	2.92	2.35	91.7	28.4	15.3
5.	2.02	72.1	126.6	96.6	—	4.05	—	68.4	25.4

W. P. S.

Fat of Momordica Seeds. C. E. Corfield and E. Caird. (*Pharm. J.*, 1920, 104, 48-44)—*Momordica cochinchinensis* is a cucurbitaceous plant indigenous to Bengal, Formosa, and the Philippine Islands. When extracted with petroleum spirit the kernels (68.3 per cent. of the seed) yielded 47.06 per cent. of a pale brown viscid oil, which on exposure to the air was gradually converted into a white solid mass. Expression of the kernels at about 40° C. yielded a pale greenish-brown fat with the following characteristics: $[n]_{D 40^{\circ} C.}$, 1.995; melting point, 28°-32° C.; acid value, 1.9; saponification value, 185.2; iodine value, 23.4; m. pt. of fatty acids, 46°-51° C.; and iodine value of fatty acids about 40. On exposure to the air the fat gradually became granular in texture owing to the effect of oxidation accelerated by the action of light. When heated to 50° C. the fat became less granular, more homogeneous and finally dark brown, and at 200°-240° C. was converted into a greenish-brown mobile liquid which remained fluid and of about the consistence of castor oil when cooled. In this form it behaved like a semi-drying oil, forming a transparent film on exposure for more than two days. The original fat consisted mainly of glycerides and contained no wax alcohols. C. A. M.

Squalene: A Highly Unsaturated Hydrocarbon in Shark Liver Oil. M. Tsujimoto. (*J. Ind. and Eng. Chem.*, 1920, 12, 63-72.)—In a previous paper (ANALYST, 1918, 43, 143) the author has shown that shark liver oil contains a high proportion of squalene, and he now gives the results of the examination of the liver oils obtained from thirty-six species of sharks. It is shown that oils of low sp. gr. (below 0.9 at 15°/4° C.) contained large quantities of squalene (up to 84.8 per cent.), whilst oils of high sp. gr. (above 0.9) did not, with a few exceptions, contain the hydrocarbon. The latter occurs more frequently in the liver oils from sharks belonging to the family *Squalidae*, but it is also present in notable quantity in the liver oils of *Scylliorhinidae*, *Chlamydoselachidae* (frill-shark), *Dalatidae*, and *Cetorhinidae* (basking-shark). Squalene has a sp. gr. of 0.8591 at 15°/4° C.; at ordinary temperature it absorbs 20.3 per cent. of oxygen from the atmosphere in four days and 24 per cent. in twenty-eight days. It readily absorbs hydrogen chloride, yielding a hexahydrochloride, m.p. 125° C. (with decomposition), which is soluble in hot alcohol, hot acetone, warm carbon tetrachloride, and cold chloroform, but almost insoluble in ether, cold alcohol, and cold acetone; it also yields a hexahydrobromide, m.p. 125° C., and a hexahydroiodide, m.p. 108° C. To estimate the amount of squalene present in the oil, 50 grms. of the sample are distilled under 10 mm. (or less) pressure; the acidity of the distillate is estimated and calculated as oleic acid, and the quantity deducted from the weight of the distillate; the difference gives the approximate amount of squalene. Considering that squalene hexahydrochloride, $C_{30}H_{50} \cdot 6HCl$, has properties very similar to those of spinacene hexahydrochloride, $C_{29}H_{48} \cdot 6HCl$ (cf. Chapman, ANALYST, 1917, 42, 161), and that spinacene has been obtained from an oil of the *Squalidae* sharks, it is highly probable that the two substances are identical. W. P. S.

Occurrence of Squalene in the Oil from Shark's Eggs. M. Tsujimoto. (*J. Ind. and Eng. Chem.*, 1920, 12, 73.)—A female specimen of the shark named

Kinbei-zame (*Lepidorhinus kinbei*) contained twelve eggs; nine of these eggs, having a total weight of 1,260 grms., yielded 218 grms. of light-brown solid fat, which contained 33 per cent. of unsaponifiable matter, including about 13 per cent. of squalene.
W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Seasonal Variation in the Chemical Composition of Marine Algæ. L. Lapicque. (*Compt. rend.*, 1919, **169**, 1426-1428.)—Studies on the *Laminaria flexicaulis* showed that whereas the appearance and consistence of the fresh seaweed were practically the same at all seasons, the dry substance varied according to the season, being 24 to 25 per cent. in the summer and only 15 to 16 per cent. in March. When dried in the air the leaves become covered with a white efflorescence, which is purely saline in the case of the spring plants, but consists of saccharine substances in the summer leaves. This efflorescence is nearly pure mannitol, but a soluble carbohydrate remains in the interior of the leaves which somewhat resembles glycogen. This is extracted by digestion with 1 per cent. acetic acid at the temperature of the water-bath. It corresponds with the laminarin of Schmiedeberg, and is abundant in the summer leaves, but present merely as a trace in the spring. The composition of the dry substance of the plant at various seasons is shown in the table below :

Season.	Soluble Carbohydrates. Per Cent.	Ash Soluble. Per Cent.	Ash Insoluble. Per Cent.
Spring equinox	1.1	28.2	7.5
End of May ...	5.0	20.1	6.3
Summer solstice	8.9	15.8	6.4
August-September	33.9	13.4	4.6

Thus, after the spring, the cell juice of the seaweeds becomes richer in laminarin and mannitol and poorer in salts, this variation being probably the result of the chlorophyllian function under the influence of increased illumination. J. F. B.

Detection and Estimation of Albumen in Urine. O. Mayer. (*Zeitsch. anal. Chem.*, 1919, **58**, 337-346.)—For the detection of albumen, the author says, reliance should not be placed on any single test; two at least should be applied, and he gives preference to the following in the order named :

1. The urine is boiled with half its volume of a saturated solution of sodium chloride and acidified with acetic acid. A positive reaction is given if as much as 0.005 per cent. albumen is present. The salt tends to prevent, but does not always wholly prevent, the precipitation of mucinoid substances. A precipitate may consist wholly or partially of resin acids, but these are dissolved by adding two volumes of alcohol, which leaves albumen undissolved.

2. The possible ambiguity of the results of the above test is removed if a portion

of the urine mixed with an equal volume of 6 per cent. acetic acid develops no cloudiness. In that case mucinoid substances are absent.

3. For this test and for the quantitative test described below the following reagent, which is substantially that of Stütz, except that the proportion of sodium chloride has been increased, is used. It contains 10 grms. mercuric chloride, 25 grms. citric acid, and 56 grms. sodium chloride in 500 c.c. The urine (5 c.c.) is mixed with an equal volume of the reagent, when the presence of 0.001 per cent. albumen occasions a precipitate; but here, again, mucinoid substances may introduce ambiguity.

4. The urine is acidified with acetic acid, and mixed with five drops of a 20 per cent. solution of sulphosalicylic acid. As little as 0.0015 per cent. albumen gives an opalescence; larger proportions a definite precipitate. Mucinoid substances do not interfere, but urine, very rich in urates, may need diluting with an equal volume of water.

Estimation.—The method depends on reaction 3 above, carried out as a zone reaction, with urine more and more diluted until the limit of sensibility is reached. To 5 c.c. of the reagent in a test-tube, 5 c.c. of the urine (or a suitable dilution of the urine if preliminary tests indicate much albumen) are added. If a strong reaction is obtained, a weaker dilution is tested in a similar manner. If the result is again positive, a still weaker dilution is tested; but if no cloudy zone appear, an intermediate dilution is tried. A table is given, showing the approximate percentage of albumen necessary to give a visible reaction within a minute when the tubes are viewed against a black ground. The method is only an approximate one, but is quickly carried out, three or four experiments sufficing, with a little experience, to obtain results of sufficient accuracy for clinical purposes. The following is an abbreviation of the table given:

Dilution.	Percentage of Albumen.
Undiluted.	0.001
1 : 1	0.002
1 : 10	0.01
1 : 100	0.1
1 : 1000	1.0

G. C. J.

Mutual Precipitation of the Toxins and their Antitoxins. Titration of Antidiphtheritic and Antitetanic Sera. M. Nicolle, E. Debains, and E. Césari. (*Compt. rend.*, 1919, 169, 1433-1434.)—Advantage is taken of the mutual precipitating property of toxins and antitoxins to afford a method for the valuation of antidiphtheritic and antitetanic sera. The success of the method depends on the employment of concentrated toxins. Filtrates from the respective cultures are saturated with anhydrous sodium sulphate. The precipitate obtained is dried *in vacuo* over sulphuric acid, ground to a homogeneous powder, and 0.8 gm. of the powder is dissolved in 10 c.c. of distilled water. Equal parts of the toxic solution and a 10 per cent. neutral solution of gelatin in physiological water are mixed

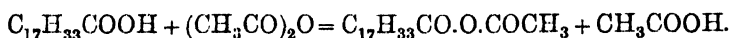
together, and 1 c.c. of the mixture is placed in each of a series of tubes. The solution is then allowed to set in the ice-box. One c.c. of antitoxic serum at increasing dilutions is poured upon the solid jellies, and these are set aside for two hours at the ordinary temperature and then examined. A positive result is indicated by a whitish disc at the junction between the diluted serum and the toxin-gelatin. It is important that both solutions should be absolutely clear. The antidiphtheritic serum is delivered at a standard of 300 units per c.c. and the antitetanic at 4,000 units. Both these standards correspond, on the above test, with the appearance of a sharp disc at a dilution of 1 : 50. The results of the method performed *in vitro* as above correspond perfectly with those obtained in standardisation on the living animal.

J. F. B.

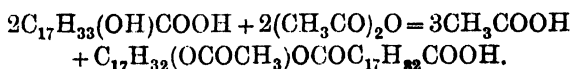
ORGANIC ANALYSIS.

Errors in the Determination of the Acetyl Value and their Remedy.

A. Grün. (*Oel u. Fettind.*, 1919, 1, 339-341, 364-367; *Chem. Zentr.*, 1919, 90, IV., 645-646; through *J. Soc. Chem. Ind.*, 1920, 39, 33A.)—The tendency of hydroxy-acids to form inner anhydrides is a source of error which is not entirely eliminated in Lewkowitsch's method of determining the acetyl value, since mixed anhydrides may be formed as in the reaction :



When such anhydrides are formed, too much acetic acid will be found by the distillation method. A more serious source of error is that caused by the formation of inner esters of hydroxy fatty acids. For example, ricinoleic acid, after eight hours' boiling with acetic anhydride, was converted, not into acetylricinoleic acid, but into acetyldiricinoleic acid :



In the case of this by-reaction the acetyl values determined by Lewkowitsch's method will be too low, whilst by Benedikt and Ulzer's method they will be too high. An objection to the ordinary methods of determining the acetyl values of neutral fats is that reciprocal esterification between the triglyceride and acetic anhydride may take place, whilst it is difficult to distinguish between the different form of hydroxylated constituents indicated by the value. To obviate these difficulties a method has been devised in which the fatty acid esters are acetylated. These esters are prepared by heating the fat with methyl or ethyl alcohol and 1 to 2 per cent. of hydrochloric or sulphuric acid, evaporating the alcohol, and neutralising, washing, and drying the residue. Neither acid anhydrides nor inner esters can be formed on acetylating these esters, which is preferably done by the original method of Benedikt and Ulzer, after removal of volatile esters. The acetyl value of free acids is obtained by multiplying the acetyl value of the esters with the theoretical acetyl value of the free acids and dividing by the theoretical acetyl value of the esters. In the case of the methyl esters, calculation into the triglyceride is unnecessary, since the difference between the group C_3H_9 and the

group C_3H_7 , is negligible. For the determination of the amount of mono- and diglycerides, the neutralised fat is acetylated and the acetic acid content of the product estimated. The difference between the acetyl value of the neutral fat and that of the mixture of methyl esters prepared from it corresponds to the amount of mono- and diglycerides. Acetylation of the fatty acid esters instead of the free acids or neutral fat is also advisable for the determination of the hydroxyl value.

Estimation of Aniline in Commercial Anilines. W. J. Sanderson and W. J. Jones. (*J. Soc. Chem. Ind.*, 1920, 39, 8T.)—A method based on the relation between the freezing-point of the sample and its aniline content is described. Owing to the solubility of water in aniline, it is necessary first to bring the sample to a definite state of dehydration or hydration. Definite hydration is effected by adding to the sample of aniline about 3 per cent. of water, which is just sufficient to saturate it, and then about 20 per cent. of solid sodium hydroxide. The mixture is allowed to stand for twelve hours or longer, with occasional shaking, after which the aniline layer is separated and its freezing-point determined by the usual Beckmann method. The commercial aniline thus treated comes to an equilibrium with the water, and the freezing-point then depends only on the aniline content of the sample. For example, pure aniline treated in the above manner holds in solution an amount of water which causes it to freeze at $-6.30^{\circ}C.$, whereas pure dry aniline freezes at $-6.00^{\circ}C.$ The thermometer used should be graduated in fifths or tenths of a degree, and verified at 0° and at $-6.00^{\circ}C.$ by means of aniline purified by repeated freezing, followed by dehydration over sodium hydroxide, and finally by distillation. In determining the freezing-point, the temperature falls at first and then suddenly rises; the highest temperature then recorded is taken as the freezing-point, t , of the sample. If there be x parts by weight of aniline per 100 parts of organic matter in the sample, and if the chief impurity is nitrobenzene, as in aniline oils for blue, then $x = 113.3 + 2.11 t$. If the chief impurities are the toluidines, as in aniline oils for red, $x = 110.7 + 1.70 t$.

J. F. B.

Fuel Value of Volatile Liquid Mixtures. G. Barsky. (*J. Ind. and Eng. Chem.*, 1920, 12, 77.)—The use of gelatin capsules for containing the sample is recommended in the estimation of the fuel value of volatile liquids by the bomb calorimeter. The capsules are made by turning in the caps of ordinary capsules, so that the concave end thus formed fitted the meniscus when the capsules are filled. The igniting wire is wound several times round the capsule.

W. P. S.

Motor Gasoline [Petrol] Properties, Laboratory Methods of Testing and Practical Specifications. E. W. Dean. (U.S. Bureau of Mines, *Technical Paper*, 1919, 214, pp. 36.)—The author states that it is now definitely established that the most untrustworthy test in vogue for gasoline is that of the average user who "tries it out" in his car. Engine tests, if conducted in the laboratory, in connection with power measurements, are still the last word in determining the utility of motor fuel, but the internal combustion motor is so complex a mechanism that it cannot be made to serve as an analytical instrument, except when handled by experts.

Properly interpreted results of an analysis in the chemical laboratory give more trustworthy information concerning the utility of a grade of gasoline than anything less than the most elaborately conducted engine-dynamometer tests or the collective experience of a large number of users.

A specification should require gasoline to be water white when viewed in a 4-oz. sample bottle, free from acid, and to comply with a distillation test, the details of which must vary with the purpose for which the gasoline is required. In some cases it may be necessary to introduce into the specification the "doctor" test, a corrosion or gumming test, a test for degree of unsaturation and a limit of vapour pressure, the last being necessary to insure safety in shipment by tank wagons, the limit suggested being 10 lb. per sq. inch.

Gasoline is rarely other than water white and rarely acid. It should, however, always be tested for acid, free or combined, and the test should be made not on the original sample, but on the residue left in the Engler flask after distillation. Carelessly refined gasoline may contain acid compounds which decompose during distillation.

The conditions under which the distillation is to be conducted must be rigidly defined. The Bureau of Mines apparatus is described and illustrated, and might be cited in an American specification. It is substantially that recommended by the American Society for Testing Materials for the analysis of turpentine substitutes, except that the thermometer used is of different range. The flask is a 100 c.c. Engler flask, and the rate of distillation 4-5 c.c. per minute. For ordinary automobile gasoline the author recommends that the temperature when 20 per cent. has distilled shall not be below 70° F., nor above whatever limit is fixed after due consideration of conditions of use; that the temperature when 90 per cent. has distilled shall not be above another limit similarly chosen; that the temperature when 50 per cent. has distilled shall not be higher than a mark halfway between the upper 20 per cent. limit and the 90 per cent. limit; and that the dry point shall not exceed the 90 per cent. limit by more than 40° C. He deprecates the introduction into the specification of an "initial" boiling temperature, as this is so difficult to determine that disagreement is likely to arise between buyer and seller, whilst the 20 per cent. (or if preferred a 10 per cent. or 5 per cent.) reading gives information of the same kind without risk of disagreement. The dry point is less often the cause of serious disagreement than "initial" readings, but too much stress should not be laid on a dry point test when the 90 per cent. test complies with the specification.

What figures should be introduced into the specification the author leaves to users, with the warning to make their specification as easy as possible with due regard to their requirements. If they make their specification more exacting than is necessary, they will perforce pay more and will be doing public disservice by making unnecessary inroads into the world's supply of gasoline. To be satisfactory, gasoline must possess sufficient volatile constituents to permit ready starting of a cold motor and the range of volatility must not exceed the limits of the vapourising power of the engine. Short of that, however, the larger the percentage of the original crude oil which can be utilised as motor-spirit, the greater the world's supply, and the less the cost per gallon.

The "Doctor" Test.—This test, although largely employed in refineries for detecting certain types of decomposable sulphur compounds, does not appear to have been published in any journal easy of access; the author acknowledges the assistance of C. J. Robinson of the Standard Oil Company in formulating the description he gives of it. About 125 grms. sodium hydroxide are dissolved in 1,000 c.c. water, 65 grms. litharge are added, the mixture shaken for fifteen minutes, allowed to stand for a day and the clear liquid (doctor solution) siphoned off. In making the test 10 c.c. gasoline and 5 c.c. doctor solution are shaken vigorously in a test-tube for fifteen seconds, a pinch of flowers of sulphur is added, shaking repeated for fifteen seconds and the mixture then allowed to settle. The quantity of sulphur used should be such that practically all of it floats on the surface separating the gasoline from the aqueous layer. If the gasoline is discoloured, or if the sulphur film is so dark that its yellow colour is noticeably masked, the test is reported positive and the gasoline condemned as "sour." If the liquid remains unchanged in colour, and if the sulphur film is bright yellow or only slightly discoloured with grey or flecked with black, the test is reported negative and the gasoline considered "sweet." G. C. J.

Estimation of *p*-Phenylenediamine. T. Callan and J. A. R. Henderson. (*J. Soc. Chem. Ind.*, 1919, 38, 408-410r.)—A stock solution of sodium hypochlorite is prepared by diluting to a litre about 50 c.c. of commercial sodium hypochlorite containing 12 to 15 per cent. of available chlorine. This stock solution is accurately titrated against $\frac{N}{10}$ sodium arsenite, using starch-iodide paper as indicator. Exactly 100 c.c. of the hypochlorite solution are diluted with about 100 c.c. of water, about 1 gm. of sodium carbonate is added, and 10 c.c. of the solution to be analysed, containing 2 to 6 per cent. of *p*-phenylenediamine dissolved in a slight excess of hydrochloric acid, are added slowly with stirring. The mixture should give a strong reaction with starch-iodide paper, otherwise the experiment must be repeated with less *p*-phenylenediamine or more hypochlorite. On the addition of the *p*-phenylenediamine, benzoquinone dichloroimide is rapidly precipitated as an almost white substance. The turbid solution is then titrated, without filtration, with $\frac{N}{10}$ arsenite, using starch-iodide paper as outside indicator, the end point being very sharply indicated by the disappearance of the blue colour on spotting out. The difference between the volume of arsenite required by the hypochlorite itself, and by the hypochlorite plus diamine, is the measure of the latter, each c.c. corresponding to 0.0018 gm. *p*-phenylenediamine. G. C. J.

Estimation of Sulphates in the Presence of Organic Sulphonic Acids. T. Callan, J. A. R. Henderson, and R. Barton. (*J. Soc. Chem. Ind.*, 1919, 38, 410-411r.)—Experiments are described which show that barium sulphate is not appreciably soluble in dilute solutions of organic sulphonic acids, and that there is little danger of barium sulphate being contaminated with co-precipitated barium sulphonates when precipitated from solutions containing organic sulphonic acids under the conditions generally employed in analytical work. Naphthalene- β -mono-sulphonic acid behaves exceptionally, however, and in its presence sulphates should be precipitated in cold, faintly acid, and highly dilute solution.

The following volumetric method is rapid and accurate. A quantity of the substance containing the equivalent of about 0.1-0.2 gm. anhydrous sodium sulphate is dissolved in water, and, if acid, is rendered faintly alkaline with ammonia containing calcium chloride to precipitate any carbonate which is filtered off. The solution is boiled until it no longer smells of ammonia. Exactly 20 c.c. of $\frac{N}{4}$ barium chloride are added, and the heating continued for a few minutes. The excess of barium chloride is then titrated with $\frac{N}{4}$ potassium chromate, using starch-iodide paper as indicator, the end point being determined by allowing a drop of the solution to fall on the test-paper so that the barium chromate remains concentrated in one spot whilst the clear solution spreads around it. A drop of very dilute hydrochloric acid is then spotted on to the paper so that the acid runs into the clear rim of the first spot. A blue colour develops within about ten seconds when excess of soluble chromate is present. An immediate production of colour indicates considerable excess of chromate.

G. C. J.

Estimation of Thiophene in Commercial Benzenes. A. Meyer. (*Compt. rend.*, 1919, 169, 1402-1404.)—In the manufacture of chlorobenzene by the catalytic action of iron turnings, the presence of thiophene has an extremely injurious influence on the activity of the catalyst, which becomes covered with a black deposit, consisting of a compound of thiophene with ferric chloride. The method described for the estimation of thiophene is a modification of that of Denigès, which is based on the precipitation of the thiophene by mercuric sulphate in the presence of methyl alcohol, the compound formed having the formula $\text{SO}_4(\text{HgO})_2\text{HgSC}_4\text{H}_4$. The reagent consists of a solution of 50 grms. of mercuric oxide in a mixture of 200 c.c. of sulphuric acid and 100 c.c. of water. The methyl alcohol always contains traces of acetone, which forms a precipitate with the mercuric salt, so that a blank determination must be made. In carrying out the test, 75 c.c. of methyl alcohol, 20 c.c. of Denigès' reagent, and 5 c.c. of benzene are placed in a stoppered bottle, and a similar mixture is made for the blank experiment, using benzene free from thiophene. The mixture is shaken occasionally during half an hour and filtered. Ten c.c. of the filtrate or of the clear supernatant liquid, if filtration be omitted, are withdrawn, diluted with 20 c.c. of water, a few drops of nitric acid free from nitrous acid are added and about 1 c.c. of iron alum solution. The mixture is then titrated with $\frac{N}{10}$ thiocyanate solution to a permanent pink. The difference between the blank titration and the main titration gives the thiocyanate equivalent of the mercury precipitated by the thiophene. One c.c. of $\frac{N}{10}$ thiocyanate corresponds to 0.01 gm. Hg, and this, multiplied by 0.14, gives the thiophene in the precipitate.

J. F. B.

INORGANIC ANALYSIS.

Estimation of Metallic Aluminium in Aluminium Dross. F. K. Bezenberger. (*J. Ind. and Eng. Chem.*, 1920, 12, 78-79.)—The dross contains a high percentage of alumina, besides metals such as copper, zinc, and iron; to estimate metallic aluminium in this mixture, advantage is taken of the fact that the metal is dissolved by bromine-water, whilst the oxide is scarcely attacked. About 1 gm. of

the dross is treated with saturated bromine-water, and a few drops of bromine are added from time to time as long as the reaction continues. The solution is then heated to expel free bromine, filtered, the insoluble portion washed, and the aluminium in the filtrate precipitated as hydroxide by ammonia. The precipitate is collected, washed, and ignited; it contains iron and copper oxides, and the quantity of these is estimated by treating the ignited precipitate with 10 per cent. sulphuric acid, which dissolves the iron and copper oxides and also a small quantity of the alumina. The copper is then deposited electrolytically, and the iron, after reduction, is titrated with permanganate solution. The sum of the iron and copper oxides thus found is deducted from the weight of the ignited precipitate in order to obtain the amount of alumina present as aluminium in the sample.

W. P. S.

Estimation of Arsenic in Tin and Tin-Plate. L. Vallery. (*Comptes rend.*, 1919, **169**, 1400-1402.)—The Marsh method gives totally erroneous results when applied to the estimation of arsenic in tin, since the formation of arsenuretted hydrogen is incomplete. The method now described is based on the distillation of the arsenic in the form of arsenious chloride in the manner usually employed in metallurgical analysis, and the estimation of the arsenic in the distillate colorimetrically after reduction to colloidal arsenic by means of hypophosphorous acid. The colorimetric method possesses advantages over volumetric methods—*e.g.*, by iodine or potassium bromate—in that the reduction by hypophosphorous acid is specific for arsenic under the conditions of the experiment, and the arsenic is produced in a visible form. The solution to be tested is compared with a standard solution in an accurate colorimeter, and the errors of the method may be minimised by matching another portion of the standard solution with the original in the same compartment of the instrument as the unknown solution. The same method may also be combined with the Marsh method for other materials by absorbing the arsenuretted hydrogen in concentrated nitric acid, then evaporating in the presence of sulphuric acid until white fumes are formed, taking up the residue with water and reducing with hypophosphorous acid. The author regards the French Government specification of 1 part of arsenic per million as too exacting in the case of refined tin, since he has generally found results ranging from 2 to 5 parts per million, and he suggests that the stringency of this specification may be due to the erroneous results obtained by the Marsh method.

J. F. B.

Estimation of Combustible Matter in Silicate and Carbonate Rocks. A. C. Fieldner, W. A. Selvig, and G. B. Taylor. (U.S. Bureau of Mines, *Technical Paper*, 1919, **212**, pp. 24.)—From 0.2 to 1 gm. of the finely ground sample is weighed with a 100 c.c. platinum dish. If the silicate rock be free from more than traces of carbonate, 15 c.c. of concentrated hydrochloric acid are added and the mixture heated on the hot plate for ten minutes. Then 20 c.c. of hydrofluoric acid are added and the contents of the dish are heated to incipient boiling until the organic matter seems to be decomposed. By conducting the digestion at temperatures not above that of incipient boiling, losses of organic carbon and hydrogen at this stage are reduced to negligible

proportions. About an hour is required for the digestion. More of the acid mixture is added from time to time to maintain the volume above 15 c.c. If concentrated much more than this there is some risk, amounting to a certainty if evaporation be pushed to dryness, that insoluble hydrated acid fluorides will form. When decomposition is complete, the mixture is evaporated to 15 c.c., 25 c.c. hydrochloric acid are added and the mixture boiled, and in order to redissolve acid aluminium fluoride an equal volume of hot water is added and the mixture again boiled and filtered hot through a small asbestos filter. This is washed free from chlorides with hot water, dried at 105° C., transferred to a combustion tube, and carbon and hydrogen are determined in the usual manner. If the sample is chiefly organic matter, the preliminary digestion is best conducted on a water-bath.

Carbonate rocks are first treated with 30 c.c. dilute (1 : 2) hydrochloric acid, the insoluble residue is filtered off on asbestos, washed with acid water, transferred to a platinum dish and then digested with hydrochloric and hydrofluoric acids and otherwise treated as described above for silicate rocks. The preliminary treatment with hydrochloric acid is necessary to avoid the subsequent formation of comparatively insoluble hydrated compounds of the type $\text{CaF}_2 \cdot 2\text{HF} \cdot 6\text{H}_2\text{O}$.

G. C. J.

Qualitative and Quantitative Test for Molybdenum in Steel and Iron.
S. L. Malowan. (*Zeitsch. anorg. Chem.*, 1919, 108, 73-80; through *J. Soc. Chem. Ind.*, 1920, 39, 27A.)—A specific and very sensitive test for molybdenum is given by xanthic acid. The test is best carried out with freshly prepared xanthate solution in the following manner: A solution of potassium hydroxide in absolute alcohol is shaken with excess of carbon bisulphide until no more of the latter is dissolved. To the solution so obtained 30 per cent. acetic acid is added until a slight yellow turbidity is formed, and the reagent is added drop by drop to the solution to be tested. If the latter has been prepared by dissolving the test material in strong acid, most of this should be neutralised before adding the reagent. If molybdenum is present, an intense red colour immediately develops, which is quite stable in water, and the intensity of which is proportional to the amount of molybdenum present. The sensitiveness of the reaction is such that 0.000005 gm. Mo in 0.0007 per cent. solution can be detected with certainty in presence of other elements. The colour is readily soluble in alcohol, amyl alcohol, ether, and chloroform, less readily in petroleum spirit and benzene. It is readily extracted from its reddish-violet ethereal solution by alkalis, and is precipitated from the alkaline solution by acids. The ethereal solution decomposes on standing, with formation of molybdenum sulphide. The reaction is not given by vanadium, tungsten, titanium, or uranium; chromates give a dark coloration with xanthic acid, and should be reduced, if present, before the test is made. The reaction can be used for the colorimetric estimation of molybdenum, by comparison with a standard solution; but, since ethereal solutions decompose too quickly, the ether should be mixed with 35 per cent. of petroleum spirit for the extraction and with 70 per cent. of petroleum spirit for the necessary dilution.

Indirect Detection of Tin. F. Feigl. (*Chem. Zeit.*, 1919, **43**, 861.)—The author briefly summarises the difficulties attendant on the detection of traces of tin in presence of antimony by the more usual methods, and recommends the following indirect method. The mixed sulphides are dissolved in hot, strong, hydrochloric acid, the antimony and tin are precipitated by means of pure zinc and then treated with strong hydrochloric acid, which dissolves the whole of the tin (which dilute acid will not do), and, if some antimony is also dissolved, it will not interfere. The solution is added to hot dilute ferric chloride solution, solid Rochelle salt or citric acid is added to prevent the subsequent precipitation of ferric hydroxide, and then an ammoniacal alcoholic solution of dimethylglyoxime is added. A red coloration proves the presence of ferrous iron and, indirectly, of tin in the original solution. As little as 0.00001 mgrm. tin may be detected in 1 c.c. G. C. J.

New Reaction of Salts of Tin. A. Mazuir (*Ann. Chim. anal. appl.*, 1920, **2**, 9)—A reaction for tin is based upon the fact that stannous and stannic iodides are completely insoluble in concentrated sulphuric acid: From 1 to 2 c.c. of the solution under examination is treated with an equal volume of a 10 per cent. solution of potassium iodide, and then shaken with 1 to 2 c.c. of concentrated sulphuric acid. In the presence of tin a characteristic yellow precipitate is immediately produced. The test is capable of detecting tin in a solution containing 0.1 to 0.2 grm. per litre. Arsenic gives a similar reaction, but the resulting arsenic iodide is completely insoluble in concentrated hydrochloric acid, whereas the iodides of tin dissolve readily in that acid in the cold. Antimony under the same conditions gives a flocculent brick-red precipitate. The metals of the antimony group do not interfere with the test. C. A. M.

APPARATUS, ETC.

Constant Temperature Still-head for Light Oil Fractionation. F. M. Washburn. (*J. Ind. and Eng. Chem.*, 1920, **12**, 73-77.)—The fractionating apparatus described consists of two parts—a Hempel column attached to the distillation flask and a constant temperature still-head. The latter is a spiral, about 6 inches in diameter, made from a 12-foot length of $\frac{3}{4}$ inch wrought-iron pipe. The spiral is immersed in an oil-bath, and its lower end is connected with the side-tube at the top of the Hempel column, whilst its upper end is provided with a side-tube which connects with an ordinary condenser. The oil-bath is heated to the required temperature by means of a coil of resistance wire immersed in the bath, and maintained at that temperature by a mercury thermoregulator. The distillation flask has a capacity of 1,500 c.c., so that 1,000 c.c. of oil may be taken for the distillation, and the flask is heated by a Rose burner with a hemispherical top covered with asbestos-coated gauze. Almost all the components of a mixture such as benzene, toluene, and solvent naphtha is obtained in a practically pure state; the intermediate fractions have each only about 1.5 per cent. of the volume of the total oil, and the compositions of these intermediate fractions are found by separate

distillations in a standard apparatus, reference being made to previously constructed curves to ascertain the percentage of benzene and toluene and of toluene and xylene present.

W. P. S.

ERRATUM.—ANALYST, *December*, 1919, p. 416, line 21 from top, for G. Hughes read J. Hughes.



GOVERNMENT REPORT.

Report of the Government Chemist upon the Work of the Government Laboratory for the Year ended March 31, 1919. Md. 419. (Price 2d. net. H.M. Stationery Office.)—The total number of samples examined in the course of the year, including those dealt with at the outpost chemical stations, was 289,180 as compared with 269,118 in the preceding year. The number examined at the central laboratory alone amounted to 184,418, the remaining 104,762 samples having been examined at various chemical stations. Partial revival of trade and the removal of restrictions on wine imports led to an increase of 42,123 samples of wine examined, mainly at the ports of entry.

CUSTOMS AND EXCISE: BEER.—The total number of samples of finished beer examined for the year in connection with duty, was 18,802. The object of the examination is to determine the original gravity of the beer before fermentation, as it is on this basis that the duty charge is raised. *Materials Used in Brewing.*—The number of samples examined was 205, of which 132 were malt, corn, brewing sugars, and exhausted grains, and 73 yeast foods and miscellaneous substances used, or proposed to be used, in the preparation of beer. In no instance was there evidence of dilution in the samples of beers taken from the premises of publicans or other retailers for the purpose of checking dilution and adulteration. Seventy-seven samples were submitted as non-alcoholic beers under Section 8 of the Finance Act (Sess. 2), 1914, which exempts from duty beers which are not brewed on licensed premises, if the original gravity and proof-spirit do not exceed 16° and 2 per cent. respectively. In four cases the proof-spirit was between 3 and 4 per cent., and in one instance it exceeded 5 per cent. Owing to the restrictions caused by the war, the importation of beer has almost ceased, and very few samples were examined. *Examination of Beer and Brewing Materials for Arsenic.*—The total number of samples tested, including beer, worts, malt, sugar, and other materials used in brewing was 1,249. Of these, eight were found to contain arsenic in excess of the limits suggested by the Royal Commission on Arsenical Poisoning—namely, the equivalent of $\frac{1}{100}$ grain of arsenious oxide per pound in the case of solids, or per gallon in the case of liquids. Of 205 samples of malt and sugar tested, none contained arsenic in excess of the limits. Nine hundred and sixty-six samples of beer and wort examined were also free from arsenic. The samples in which excess of

arsenic was found consisted of salts and solutions proposed to be used for treating brewing waters, but in no case was arsenic, in excess of the Royal Commission limits, found in samples of beer made with water so treated.

CIDER AND PERRY.—Eighteen samples of imported cider were examined, all of which were of normal character. Ten samples were purchased by Customs and Excise officers in the United Kingdom, of which four were factitious—that is to say, did not consist solely of fermented apple-juice. The six genuine ciders contained 3 to 6 per cent. of proof spirit. Six samples of sour ciders were also submitted for examination in order to obtain remission of the duty. All were found to contain sufficient acid to render them unsaleable as commercial ciders.

TABLE-WATER DUTY.—The Finance (New Duties) Act, 1916, and the Finance Act, 1916 (6 Geo. V., c. 2, and 6 and 7 Geo. V., c. 24), imposed Excise and Customs duties on "Table Waters" at rates varying from 2d. to 8d. per gallon. In order to determine the question of liability to duty, six samples of mineral and spa water, 135 samples of cordials, syrups, fruit juices, and essences, and forty-two samples of "non-alcoholic" wines were examined. Of the six waters, five proved to be medicinal, and one was liable to table-water duty; twelve of the so-called "non-alcoholic" wines contained more than 2 per cent. of proof spirit, and one herbal preparation, which the vendors claimed was not a beverage, but a *bona-fide* medicine, proved to be sufficiently medicated to exempt it from table-water duty.

SPIRITS.—*Illicitly Distilled Spirits*: Thirty-one samples of spirits suspected to have been obtained by illicit distillation were examined during the year, and in fifteen instances strong evidence of the presence of illicit spirit was obtained. The latter samples were taken from two sets of premises, where, on investigation, it was evident that illicit distillation had been carried on. *Imported Spirits and Spirituous Preparations*: The number of samples examined was 11,444, a further decrease on last year's numbers, though in spirits, as distinct from spirituous preparations, the number of samples was doubled. This was no doubt due to the fact that a shortage of spirit existed up to the date of the Armistice, shipping space being found, however, for the pure spirit required in such large quantity in connection with the production of munitions. When necessary, the amount of sugar, saccharin, and other dutiable ingredients in the imported preparations was also determined.

More than 1,000 papers were dealt with relating to applications received by the Commissions of Customs and Excise, either (1) in respect of claims for rebate on alcohol used in making medicinal preparations; or (2) in connection with permission to receive methylated spirit and other forms of duty-free alcohol for use in manufactures, tuition and research. In the former instances advice was required as to whether the claims in question were such as could properly be allowed under Section 4 of the Finance Act, 1918.

WINES.—The number of samples examined in connection with the assessment of the Wine Duties was 84,796, including a few samples of unfermented grape-juice and medicated wine. In addition to the samples of so-called wine dealt with as table-waters, one sample of medicated wine and nine of British wines or sweets

were also examined to settle questions of exemption or of liability to licence duty in connection with sales. Of ordinary wine, there was a very largely increased importation, more than 40,000 additional samples having been examined, for the most part after the date of the Armistice.

TOBACCO.—*Manufactured Tobacco for Home Consumption*: To ensure observance of the regulations as to moisture and oil, which are based upon the provisions of the Customs and Inland Revenue Act, 1887, the Oil in Tobacco Act, 1900, and the Finance Act, 1904, samples are taken from the factories and from the premises of retail dealers by Customs and Excise officers, and examined at the Government laboratory. *Manufactured Tobacco and Commercial Snuff on which Drawback is claimed on Exportation*: The samples of tobacco exported on drawback numbered 27,123, as compared with 30,564 received during the previous year. The volume of the export trade in commercial snuff is small, the number of the samples of this kind received being only 133, or 20 less than in the year 1917-18.

SUGAR, GLUCOSE, AND SACCHARIN.—*Sugar*: The variety of preparations containing sugar is so great that it has been found necessary to adopt fixed rates of duty in the case of those which are regularly imported or which are exported on drawback, samples being examined only in cases of doubt as to their description or rating for duty purpose. *Glucose*: Glucose manufactured in this country is subject to an Excise duty. In connection with the assessment of this duty 266 samples of glucose solution taken from the charging vessels during the course of manufacture were examined in order to check the manufacturers' declarations, and 42 for assessment of drawback on exportation, together with 73 of glucose used in syrups made at sugar refineries. *Saccharin*: The manufacture of saccharin in the United Kingdom, which had ceased for some years, was resumed in 1917, and 165 samples of saccharin and of the materials used in its production were examined in connection with the assessment of duty.

TEA.—During the year under revision the inspection of tea on importation was undertaken by the Ministry of Food. Only in the case of tea which had not passed into consumption for any reason (*e.g.*, broken packages, damaged tea, etc.) were samples committed to the Government laboratory. Hence the high proportion of samples rejected as unfit for human consumption, viz., 4,862 out of a total of 7,987 samples submitted, or approximately 60 per cent. The quantity of tea represented by these samples was insignificant compared with the total quantity passed by the Tea Controller for home consumption.

MATCHES.—The imports of matches in 1918-19, though larger than in the previous year, were still much below pre-war level, and only three samples were submitted under the "White Phosphorus Matches Prohibition Act, 1908," in none of which was any evidence of the presence of white phosphorus discovered.

BOARD OF AGRICULTURE AND FISHERIES.—The number of samples examined for the Board of Agriculture and Fisheries, the Board of Agriculture for Scotland, and

the Department of Agriculture and Technical Instructions for Ireland, was 581 as compared with 1,568 in the previous year. Samples of imported dairy produce and margarine are taken at the ports of Customs and Excise officers in accordance with the provisions of the Sale of Food and Drugs Act, 1899, and the Butter and Margarine Act, 1907. The number includes the samples taken under the Public Health (Milk and Cream) Regulations, 1912 (see Local Government Board—Preservatives). *Imported Milk and Cream.*—During the past year no samples of fresh or of sterilised milk were examined. The description “condensed milk” is usually applied to milk from which water has been removed by evaporation to such a degree that one part by weight of the condensed product contains the milk solids of two and a half to three parts of the original milk. This condensed milk is liquid, and, if it has not been sweetened, contains from 64 to 68 per cent. of water. It frequently, however, contains 40 per cent. of sugar with only 24 to 29 per cent. of water. The condensed unsweetened milk prepared in America is usually described as “evaporated” milk. This consists of milk which has been evaporated to about half its bulk, and one part by weight therefore contains the milk solids of only two parts of the original milk. The proportion of water is from 74 to 77 per cent. Of late years condensed milk has also been prepared in which the removal of the water has been carried much farther, leaving the milk solids in a nearly dry condition. This product, which is variously described as “dried milk” or “milk powder,” contains the milk solids of seven or eight times its weight of whole milk, or, when prepared from skimmed milk, the milk solids of nine to ten times its weight of skimmed or separated milk. The water in “milk powder” varies from 2 to 8 per cent. The object of the examination is to ascertain whether the milk has been prepared from impoverished, skimmed, or separated milk, in which cases the product can only be imported in receptacles marked in accordance with the requirements of the Food and Drugs Act. The samples are also examined for preservatives (ANALYST, 1918, 43, 239). Of 51 samples of condensed, sweetened, and unsweetened milk, 47 were condensed whole milk, and 4 condensed skim milk. Three of the packages were not marked as required by law. The percentage of fat in the samples of condensed whole milk ranged from 7.2 to 9.9. The samples with the low percentage of fat were taken from consignments of “evaporated” milk, 30 of the 47 samples consisting of this product. All the samples were free from preservatives, and there was no evidence that the samples of condensed whole milk had been prepared from impoverished or skimmed milk. No samples of dried milk were examined; no samples of cream were received during the year. *Imported Butter.*—Only 20 samples of imported butter were examined as compared with 431 in 1917-18 and 892 in 1916-17. Of the 20 samples, 14 were from Denmark, 4 from the United States, 1 from Australia, and 1 from the Argentine. The samples were examined in every case as to the purity of the fat and the presence of boric acid and added colouring matter. The water was also determined, but no sample was found to contain water in excess of the legal limit of 16 per cent. There was no evidence of the presence of any other fat than butter in any of the samples. *Imported Margarine.*—No samples of imported margarine were examined. *Imported Cheese.*—Of the 12 samples of imported cheese examined

all proved to be genuine. *Questions submitted by the Board for Report.*—In the course of the past year numerous matters have been submitted by the Board for report. Many of these related to the value of materials supplied as feeding-stuffs, and to the supply of potash and phosphatic manures. The official method for the determination of potash in fertilisers has been revised and a new method drawn up for use in the case of flue dust (F. and F. Stuffs [Methods of Analysis] Regulations, No. 659, 1918). Reports on Paisley meal, butter-milk, and fish meal, have been made to the Board of Agriculture for Scotland.

A description is given of the work done for the Fisheries Division, Central Control Board (Liquor Traffic), Crown Agents for the Colonies, Food Ministry, Home Office, India Office, Inland Revenue, Local Government Board, Ministry of Munitions, Post Office, Public Record Office, Stationary Office, Board of Trade, Coal Controller, War Department and Office of Works.

Under the Local Government Board are mentioned samples of chocolates containing mercury derived from the tin trays in the process of manufacture, and a case of pastry made with a self-raising flour in the preparation of which tartar emetic had been used in the place of tartar substitute (acid phosphate of lime).

SALE OF FOOD AND DRUGS ACT.—Examination of samples referred by Magistrates under the Sale of Food and Drugs Acts of 1875 and 1899, and under Food Control Orders.—When proceedings are taken under these Acts, either the prosecutor or the person charged may request the justices before whom the case is heard to forward a portion of the sample in question to the Government Laboratory for analysis. Under these provisions 97 samples were received as compared with 102 in the previous year. These included 82 samples of milk; 3 of whisky; 2 each of ginger, Epsom salts and dripping; and 1 each of lard, cheese, chocolate, beer, camphorated oil and sausages. Five samples could not be examined owing to their condition on receipt. The results of the examination differed from those of the Public Analyst in 12 out of the 92 cases, or 13 per cent., as against 13·5 per cent. in the previous year. The disagreements in respect of milk samples were 5 out of 82. The following are the particulars of some of the most important cases: *Whisky.*—The results as to the strength confirmed those of the Public Analyst in 2 cases, but differed in 1 case. *Epsom Salts.*—One sample contained arsenic, and the other was impure, as alleged by the Public Analyst. *Dripping.*—The results showed that both samples, which were alleged to be tallow, came within the description of dripping as defined by the Food Control Order. In the case of ginger alleged to contain foreign starch, chocolate alleged to be deficient in cocoa powder, beer alleged to have a low original gravity, the results did not agree with those of the Public Analyst. In the case of camphorated oil alleged to be deficient in camphor, lard alleged to be beef or mutton fat, cream cheese alleged to contain boric acid, and sausages alleged to be deficient in meat, the results agreed with those of the Public Analyst. In the 77 cases in which certificates were issued, the charges were as follows: In 44 cases, added water; 22, abstraction of fat; 8, added water and abstraction of fat; 2, presence of pre-

servatives ; and 1, presence of dirt. Boric acid was also alleged to be present in one of the samples.

FERTILISERS AND FEEDING STUFFS ACT.—The five fertilisers examined consisted of superphosphate, basic slag and sulphate of ammonia. The six feeding stuffs consisted of feeding meals and cakes, milling by-products and poultry foods. Several of the meals contained a considerable proportion of substances unsuitable for feeding purposes.

H. F. E. H.



REVIEW.

THE BACTERIOLOGY AND MYCOLOGY OF FOODS. By FRED WILBUR TANNER, M.S., Ph.D.
New York : John Wiley and Sons. 1919. Pp. 592. London : Messrs. Chapman
and Hall, Ltd. Price 28s.

In this treatise the author has attempted to cover a very wide field, and one in which there is much room for improvement in both knowledge and method. After eight chapters dealing with the technique of bacteriological analysis and the general description of yeasts, moulds, and bacteria, and the chemistry of food materials, there follow chapters on the bacteriological examination of Air, Water, Milk and its Products, Eggs, Meat and Meat Products, Canned Foods, etc. The work is almost entirely a compilation from various sources, chiefly American, and though in the preface the author states that the methods of analysis have been presented together with a discussion based on the literature of the subject, this discussion is of the nature of an epitome of the views of others rather than a presentation of the author's own opinion on the various subjects.

In these days, when the volume of scientific literature is becoming so large, it is true that an accurate compilation is often of great value; but it is absolutely necessary that such a compilation should be very carefully made, and it is disappointing, after reading with pleasure in the opening paragraph of the Preface that "a thorough training in chemistry is essential for intelligent work in microbiology"—a sentiment which the reviewer must cordially echo—to find that the chemist's sense of accuracy and attention to detail is singularly lacking in the reproduction of the subject-matter.

For instance, the author has accumulated in one chapter practically every medium which has been proposed for the cultivation of micro-organisms, but no attempt has been made to present them in a form which would render their preparation reasonably possible in many cases, and the necessary context of the original has too often been omitted. As an example the preparation of malachite green media (p. 52) may be quoted :

1. Three grms. Liebig's Meat Extract.
2. Acidify this with 7.5 c.c. $\frac{N}{2}$ HCl.
3. Dissolve 30 grms. of agar by boiling.
4. Neutralise with 7 c.c. $\frac{N}{2}$ NaOH until neutral to litmus.
5. Add 5 c.c. $\frac{N}{2}$ Na₂CO₃ and heat in an Arnold for several hours. A somewhat difficult proceeding!

In the directions for the preparation of media the words "slightly alkaline" or "absolutely neutral" often occur, and such terms should have no place in any modern method of preparation. In some cases the reaction is given as +1, +0.5, etc., but nowhere are any directions given for the determination of this reaction, nor is any explanation of the symbols vouchsafed.

The same faults appear in the sections dealing with methods of staining. A very fine collection of methods has been very inadequately reproduced. For such an excellent method as the Indian ink method the reader is simply directed to "mount in Chinese ink," and, though the author states that he has secured the best results for flagella staining with Loeffler's method, and gives the composition of both mordant and stain, he entirely omits to use the former in his description of the staining process. Other instances of this carelessness are only too common. The chapters on water and milk examination contain much useful information, together with full descriptions of the American official methods, but a good deal of space is allotted to chemical analysis, which hardly appears to come within the scope of the work.

The section dealing with eggs includes an account of work which has hitherto been hidden away in official bulletins, and this has been carefully collected here, but the coloured plates in this section have suffered in reproduction, and comparison with the explanations in the texts shows that they call for much imagination on the part of the reader.

There is no doubt that there is a great deal of information contained in this work, but it has not received the consideration it deserves in its reproduction, and at the same time there is much which, however useful, has little or no bearing on the subject-matter of the book. An illustration of this is afforded by an appendix of tables of figures, some of which are quite in their proper place, but with them are included those which give areas and circumferences of circles, water pressures at different heads, etc., together with such unusual information as the relation between Kokus and Bedeps, the number of square miles in a township, etc.

The wording of the book is decidedly American, but this should not be carried to such an extent as to take liberties with such revered names as those of Twaddell.

The text is much marred by typographical errors, which are undoubtedly the result of careless proof-reading.

To the expert reader this work may be of interest, as the author has certainly collected together a great deal of information, the value of which is enhanced by the bibliography (chiefly American) attached to each chapter, but to the student the unreliability of the text renders it an unsafe guide.

CECIL REVIS.

A. R. I. P. U. B. A.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

ORDINARY MEETING, MARCH 3, 1920.

HELD at the Chemical Society's Rooms, Burlington House, Mr. Alfred Smetham, President, in the chair.

Certificates were read for the first time in favour of Mr. Laurence Harry Mills, B.Sc. (Birmingham), A.I.C., and Dr. Frederick Robertson Dodd, F.I.C.

Certificates were read for the second time in favour of Messrs. Henry Atlas, John Carmichael, F.I.C., Herbert John Evans, B.Sc., F.I.C., Cornelius Durham Garbutt, Douglas Henville, F.I.C., Maurice S. Hutchinson, B.Sc., Benjamin Richards James, F.I.C., Geoffrey Martin, D.Sc., Ph.D., F.I.C., Robert Leitch Morris, F.I.C., and James Wood, M.A., B.Sc., F.I.C.

Mr. William John Read, M.Sc., F.I.C., was elected a Member of the Society.

The following papers were read: "The Detection of Finger-prints on Documents," by C. Ainsworth Mitchell, M.A., F.I.C.; "Photo-micography with Simple Apparatus," by T. J. Ward; and "Note on the Solubilities of Theobromine," by Raymond V. Wadsworth.

* * * * *

ESTIMATION OF FIBRE IN FEEDING STUFFS.

A conference was held in London on December 8, 1919, at which representatives of the Agricultural Education Association and the Society of Public Analysts and other Analytical Chemists discussed the estimation of fibre in feeding stuffs. The Agricultural Education Association was represented by Messrs. J. Golding, H. T. Cranfield, C. Crowther, B. F. Davis, F. V. Dulton, W. Godden, H. A. D. Neville, and G. S. Robertson, and the Society of Public Analysts and other Analytical Chemists by Dr. S. Rideal (Chairman of the Conference), Messrs. E. R. Bolton, B. Dyer, J. Hughes, E. W. Voelcker, and J. A. Voelcker.

Although no definite decisions were arrived at yet the proceedings were not without value, and the matter is still open for further discussion. The Secretaries of the two bodies concerned are collaborating with the object of arranging a scheme of investigation which may be carried out under the ægis of the Society's "Analytical Investigation Scheme."

THE DETECTION OF FINGER-PRINTS ON DOCUMENTS.**By C. AINSWORTH MITCHELL, M.A., F.I.C.***(Read at the Meeting, March 3, 1920.)*

THE study of finger-prints, which began as a scientific investigation, has developed in practice into the most trustworthy method of identifying human beings. Although the general adoption of the system by the Police Authorities all over the world has had the effect of bringing the official methods of comparison, measurement, and classification of finger-prints to a high pitch of efficiency, it has, at the same time, tended to stereotype those methods, and to discourage experiment in other directions. The result has been that many questions connected with the subject have been left



FIG. 1.—PRINTS OF SIR WILLIAM HERSCHEL'S RIGHT THUMB IN 1861 AND 1914.

to be solved by independent workers, and this applies more particularly to the detection of latent prints on documents.

So much has been published upon the individuality of finger-prints and of the methods of comparing an unknown mark with a known finger-print, that it is hardly necessary at this stage to do more than refer to the bibliography at the end of this paper. I might, however, mention the accurate method of comparison devised by Mr. A. S. Osborn, of New York, in which the resemblances between two prints are clearly shown by means of photographs beneath little squares accurately ruled upon glass.

The Kew micrometer, as suggested by Dr. Faulds, affords an accurate means of measuring the lines and position of the pores in finger-prints, and also is a most useful instrument for many other purposes in the laboratory.

Another point which may be mentioned here is the more recent evidence of the

persistence of the characteristics of the papillary ridges on the fingers of an individual throughout a long life.

I am indebted to Sir William Herschel for permission to reproduce these prints (Fig. 1), one of which was made in 1861, and the other sent to me by him in 1914, with a repeat in 1916, not long before his death. Apart from the weathering of the ridges, due to age, the patterns of the two prints agree in all essential details.

It was Sir William Herschel who first introduced the use of finger-prints as a safeguard upon documents, but the first investigation of the subject in all its bearings, and especially of accidental finger-prints as a means of identifying an unknown individual, was made by Dr. Henry Faulds, whose work has been somewhat strangely neglected. His experience affords another instance of two men, unknown to each other, studying a subject from different points of view; and so when Faulds published his work he had the misfortune to find that the main idea had been anticipated.

In the course of his investigation Faulds paid considerable attention to the special branch of the subject, which forms the subject of this paper, and suggested the use of certain reagents, to which I shall refer presently.

The points upon which I found that there was little information available were: (1) Which is the most suitable method for detecting latent finger-prints upon paper, such as that of a book, under varying conditions? (2) After what period of time is it possible to detect such prints upon different qualities of paper by the different methods?

The nearly invisible marks which are made even by heavy pressure of fingers upon, say, the leaves of a book cannot, as a rule, be shown up directly by photography with oblique illumination, as is not infrequently the case with the prints made upon polished furniture or the like, and some method of development is therefore necessary.

MECHANICAL DEVELOPMENT WITH POWDERED SUBSTANCES.

The official English method of intensifying marks made upon various objects, such as glass bottles, window-panes, woodwork, etc., is to dust the surface very lightly with a light or dark powder, according to the conditions, and to remove the excess of powder by means of a soft brush. A mixture of chalk and mercury, the "grey powder" of the pharmacist, is used for dusting dark surfaces, whilst finely powdered graphite is used for light-coloured surfaces (E. R. Henry, "Classification and Uses of Finger-Prints," p. 106).

Faulds found that a mixture of fine graphite and lampblack sold by Conté gave excellent results (H. Faulds, "Guide to Finger-Print Identification," p. 65). Other powdered substances used for the purpose are fine ferric oxide, magnesium carbonate, and aniline dyestuffs. For example, Stokis ("Annales Soc. Méd. Leg. Belge," 1906, p. 7), recommends the use of lycopodium powder with 10 per cent. of Soudan Red III., which is dusted on through a fine metal sieve, and then shaken off the paper.

Locard (*La Poroscopie*, p. 11) uses finely divided red lead, previously heated, for light surfaces, and lead carbonate for dark surfaces. He has also found lead

iodide to be effective, although its yellow colour is a drawback for photography. The advantage of heating the powder is that it will be thoroughly dry, and so not adhere to other parts of the surface, but only to the filmy impression.

For developing latent prints on paper I have found methylene blue to be an efficient agent, provided it is quite dry, and is lightly dusted over the surface and then blown off.

The results which this mechanical method is capable of yielding depend very largely upon the absorptive capacity of the paper. If the surface is very highly glazed, so as to be comparable with the surface of a japanned tin box, the prints seem to be quite permanent, and I have developed them quite sharply with methylene blue after a lapse of three years. But prints made on highly sized clayed paper, such as is used for book illustrations, although they could be brought out distinctly after several weeks, did not appear when treated in this way after three years. In the case of tracing-paper the marks were developed clearly after three weeks, but could not be brought up after a few months, whilst the prints on an ordinary card showed all their details for the course of a few days, but yielded only a faint outline after eighteen days. Similar impressions on white blotting-paper could be developed with sufficient detail for identification for an hour or two after being made, but showed only faint smudges, with no details, after about a week.

To protect the prints from being smudged after development, Stockis (*loc. cit.*) recommends coating the surface with a film of a solution of gum arabic, alum, and formaldehyde.

LIQUID CHEMICAL REAGENTS.

Forgeot (*Archives de l'Anthropol. Criminelle*, 1891, 6, 387) describes experiments made by Aubert with special papers saturated with various salts, such as silver nitrate and mercuric nitrate, the prints being subsequently developed with appropriate reagents. When the hand is pressed on white paper and subsequently developed with 10 per cent. sodium thiosulphate solution containing a few drops of alcohol, the imprints are frequently developed as oily stains. The success of these methods, however, depends upon the time during which the hand was in contact with the paper, and so they cannot be recommended as reagents for the detection of accidental finger-prints.

Osmic Acid.—Since imprints left by the finger invariably contain traces of organic matter, notably fatty compounds, the use of a solution of osmium tetroxide (osmic acid) suggests itself as a suitable reagent, and Faulds (*Dactylography*, p. 64) and Gross ("Handbuch der Untersuchungsrichter," p. 598) have both mentioned the possibility of using this reagent, but neither has given any particulars of the results which it gives.

From my experiments I find that, by brushing the ordinary 1 per cent. solution over a print and leaving the surface moist, preferably while exposed to sunlight, reduction takes place and the ridge becomes black through the reduced osmium, while the furrows remain uncoloured.

The method is capable of showing up prints distinctly on moderately sized paper after the lapse of several weeks, but is less trustworthy than some of the other

methods for long periods. Naturally it will not be effective in the case of prints on absorbent paper.

Ink Methods.—This objection also applies to the method of development by ink, which was first devised by Forgeot (*loc. cit.*), for the successful application of which a contact between the finger and paper of a few seconds at most is necessary. This method is based upon the principle that there is normally a slight secretion of oil on the fingers, and that in the impressions produced by them some of this oil will have been conveyed to those parts of the paper with which the ridges of the patterns have come in contact. Hence, when the place is subsequently treated with ink, the ridge markings will repel the liquid, whereas the intermediate furrows and the rest of the paper will be stained. In this way a negative finger-print will be produced as opposed to the positive prints given by adhering powder or osmic acid, in which the ridges receive most of the pigment and the furrows remain relatively uncoloured.

Forgeot found that the best method of applying the test was by means of a brush, and that there was no advantage in diluting the ink. In his experience developing the print in an ink-bath was fatal to good results, as it produced unequal staining.

Faulds (*loc. cit.*, p. 67), however, discarded Forgeot's method of brushing for development in a bath, and also found that ink diluted with water gave much better prints than the ink by itself.

It is highly probable that the cause of these discrepancies lies in the fact that writing ink is not a definite chemical composition, but varies widely in the proportion of its constituents. For instance, I have shown (ANALYST, 1908, 33, 8) that the amount of total solids in twenty-five different samples of commercial writing inks varied in their proportions from 1.89 to 7.94 per cent., whilst copying inks (with which Faulds also obtained good results) contain a still higher proportion of solid matter.

If comparable results are to be obtained by this method, it is necessary that a standard ink should be used, and the composition of this should be as simple as possible.

The drawback to the use of an ordinary iron-gall ink is that it is applied to the paper in an unoxidised condition, so that even those parts which appear very pale at first may eventually become much darker, with the result that it is difficult to control the final coloration. I have, therefore, made experiments with other ink compositions.

Schiff (*Ann. Chem. Pharm.*, 1871, 169, 164) attributed the blue coloration given by ferric salts with gallic and gallotannic acids, phenol, etc., to the presence of hydroxyl groups, and concluded that the intensity of the coloration depended upon the number of free hydroxyl groups in the organic compound. Schiff's work was extended by Schlüttig and Neumann (*Die Eisengallustinten*, 1890, p. 16), who established the fact that to produce a permanent coloration with iron salts—*i.e.*, an ink—a substance must contain three hydroxyl groups in juxtaposition. For example, hydroquinone with two hydroxyl groups does not yield an ink, whereas gallic acid and pyrogallol with three adjacent hydroxyl groups do form them. I have pointed

out (ANALYST, 1903, 28, 146) that this law of atomic grouping also applies to the permanent colorations produced by ammonium vanadate with similar compounds, and I have also found that the same rule is applicable to the colour reactions of osmium tetroxide. The ordinary 1 per cent. solution of osmic acid produces inks with gallotannic acid and gallic acid, which have the advantage over iron-gall ink of attaining their maximum intensity of colour almost immediately after application to the paper. The gallic and gallotannic acids form violet-grey colorations, but the most suitable ink for developing finger-prints is obtained by adding pyrogallic acid to osmic acid solution. In very dilute solution the osmic pyrogallate is at first violet,



FIG. 2.—PRINT DEVELOPED AFTER THREE YEARS WITH "SWAN" INK.



FIG. 3.—PRINT DEVELOPED WITH OSMIUM PYROGALLATE.

but almost immediately becomes greenish-blue, and when applied to paper gives a rich violet-black coloration.

This reaction may be used as a very sensitive test for osmium tetroxide, the distinctive blue coloration being obtained on adding a trace of solid pyrogallic acid to a solution containing a few drops of 1 per cent. osmic acid solution. Ink of a suitable dilution for treating finger-prints may be made by diluting 2 c.c. of the ordinary microscopic osmic acid reagent with the same quantity of water, and adding 0.05 gm. of pyrogallic acid. The liquid is ready for use immediately, and should be brushed over the paper with one broad sweep of a soft camel-hair brush.

These ink methods are applicable under varying conditions, and I have developed prints on highly sized paper after the lapse of three years both by means of ordinary "Swan" ink (total solids, 2.98 per cent.) and by means of osmic pyrogallate.

REAGENTS IN THE FORM OF VAPOUR.

The fact that finger-prints may, and probably usually do, contain a certain amount of fat suggested the use of reagents which are absorbed by fat. After I had made experiments for some months with iodine vapour, I found that its use had already been suggested many years ago by Aubert and Coulier, although no details are given as to its effectiveness as a reagent.

On the whole, it is the most sensitive reagent of any I have tried. For example, a print made with light pressure on well-sized paper could not be developed with methylene blue a month later, but was shown up sharply by means of iodine vapour, and was again redeveloped after three and a quarter years. A print on parchment paper, however, could not be developed after a few months, whilst prints on porous paper only gave moderately good results after a few hours.

The drawbacks of the iodine method are that the prints are fugitive and, owing to their colour, are difficult to photograph. They may be fixed to some extent by treatment with solutions of silver salts, followed by thiosulphate, but lose a good deal of their sharpness in the process.

I have found that prints made with mercuric iodide vapour are considerably more stable. They are obtained by exposing the paper to vapour from a heated mixture of 4 parts of mercury and 5 parts of iodine. At first they are dark red, but the colour soon changes to yellow, probably through the formation of the yellow modification of mercuric iodide. An objection to this method is that the paper itself is sometimes stained a dark colour.

Bromine vapour only produces faint yellow prints, whilst iodine chloride vapour gives a much fainter coloration than iodine. Mercuric bromide is much less satisfactory than mercuric iodide as a reagent.

Since osmium tetroxide boils at about 100°C ., it seemed probable that, applied in the form of vapour, it might prove a more sensitive reagent than when used as a solution. I found this to be the case, and obtained prints full of fine detail by holding the paper over a basin in which a little of the 1 per cent. solution was boiling. The method will give good permanent prints on moderately sized paper or a postcard, but is not so sensitive as the iodine method. It effected no development of a print on highly sized paper after a period of three years. The prints produced by osmium tetroxide vapour are positive, the ridges being grey, the furrows white, and the pores opening on the ridges dark grey to black.

Incidentally it may be mentioned that the method affords another test for osmium tetroxide. A slightly greasy finger-print is made on glazed paper, which is then exposed to the vapour of the boiling liquid. A permanent print of the type described indicates the presence of osmium tetroxide.

It is probable that ruthenium tetroxide or volatile ruthenium salts, which also darken on contact with organic substances, would also give good results in the development of latent finger-prints.

Faulds, in his experiments with the ink method, found that good results could often be obtained by adding creosote to the ink. This causes the reagent to run very rapidly over the surface of the paper, so that the period of contact is much

shorter than when ordinary ink is used. The differential staining is more pronounced when a finger-print is recent than after it has been made for some time, and Faulds was able in this way to make some estimate of the approximate age of a print.

It is obvious that any such deductions from the behaviour of a print would only be possible if all the conditions, such as the behaviour of different kinds of paper, duration of impression, and effect of temperature upon the permanency of the prints, were known.

The comparative results obtained with reagents in the different forms of powders,



FIG. 4.—RECENT PRINTS DEVELOPED WITH OSMIUM TETROXIDE VAPOUR.

liquids and vapours, appears from the results obtained in my experiments to be more likely to afford a promising basis for estimating the relative age of finger-prints.

EXAMINATION OF THE PORES.

The osmic vapour method is particularly suitable for determining the relative position, number, sizes, and shapes of the pore openings in finger-prints. This recent development of the finger-print system is due to Dr. Locard, head of the Police Laboratory at Lyons, and I am indebted to him for the examples of the prints shown on the screen illustrating this new method of examination, which he terms "poroscopy," applicable to any part of the hand. In his communication on the subject (*loc. cit.*) he states that the pores are as persistent as the ridges themselves. In this connection it is interesting to compare the prints which were sent to me by Sir William Herschel. Making every allowance for defective printing in the later print, I found that although the ridges were obviously in agreement, it was difficult to trace correspondences between the pores except in one or two instances. The weathering of the skin on the fingers in old age thus appears to interfere with the pores, at all events in the case of prints made with printing ink, to a greater extent than it does with the ridges.

The chemical method which Mr. T. J. Ward has accidentally discovered offers many advantages over the old printing-ink method for the preparation of comparative prints.

In this method an ordinary slow gelatin photographic plate is exposed to light for a few seconds; the finger is then well moistened with any usual photographic developer, and pressed firmly and evenly upon the surface of the plate for about fifteen seconds. The plate is then left to develop spontaneously, and is finally fixed with sodium thiosulphate and washed in the usual way. The image obtained shows the ridges white and the furrows and pores dark, with the finest detail clearly defined.

The difficulty in preparing or developing finger-prints so as to show the pores effectively is that the pigment used, whether in the form of printing ink or of a powder or liquid, is liable to hide the distinctive features. Referring to this drawback, Wilder and Wentworth remark in a book recently published on the subject of "Personal Identification" (p. 309): "The authors look ahead to the time, not far distant, when all accidental impressions will be developed by chemical fumes, or by other equally delicate methods, thus reproducing the impressions exactly as they are, without the intervention of any kind of powders so much in use to-day."

I trust that the results of the experiments which I have brought before you this evening may contribute towards the solution of this problem.

The great assistance which Mr. Ward has given me in photographing various prints, developed by different methods, and in preparing lantern slides from them, has enabled me to demonstrate these results to you much more effectively than I could otherwise have done, and I take this opportunity of thanking him for the interest he has taken in this work.

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PHOTOMICROGRAPHY WITH SIMPLE APPARATUS.

By THOMAS J. WARD.

(Read at the Meeting, March 3, 1920.)

RECORDS obtained by photomicrography are frequently of great service in many industries and scientific investigations at the present day, but it is a common misconception that in order to obtain satisfactory results it is necessary to employ expensive apparatus such as those figured in the catalogues of microscopical dealers. My purpose is to demonstrate that by means of simple and inexpensive apparatus it is possible to obtain satisfactory results with magnifications not exceeding about forty diameters.

The essential apparatus consists of a camera, a lens, a support for the object, and some source of illumination. The camera may be of any form, however simple, provided that it allows of extension from about 10 cm. to not less than 50 cm., and is so arranged that focussing may be achieved by altering the position of the back. Its size should allow the use of half plates if necessary. The lens may consist of any form of short focal length camera lens or a microscope objective, according to the magnification required. The micro-lenses I chiefly employ are achromatic objectives ranging in focal length from 76 to 8 mm., and costing ten shillings each in pre-war days. The object holder may be of any simple form and should be fitted with rubber bands or metal clips. Daylight in this country, at least, is too variable and uncertain to be of much use for this work, and some form of artificial illumination should be employed. I have at various times used the following: Oil lamp, batswing gas burner, incandescent gas (upright), acetylene (cycle lamp, flat flame), electric $\frac{1}{2}$ watt 50 c.p., and magnesium ribbon. Of these, the two latter are the most satisfactory for constancy, high actinic power and convenience.

The relative actinic power of the above illuminants when using ordinary (not ortho- or pan-chromatic) plates is approximately as follows:

	Equivalent Exposures.
Batswing burner	40 seconds.
Incandescent gas	10 "
Acetylene (flat flame)	6 "
Electric $\frac{1}{2}$ watt 50 c.p.	3 "
Magnesium ribbon	1 " (1.5 cm.)

PLATES.—In regard to the numerous makes upon the market there is but little to choose between them for this work. On the whole, an "Ordinary" plate is slightly superior owing to its fineness of grain and ease of manipulation, but where economy of time is a consideration one requiring shorter exposures, such as a "Special Rapid," will be found satisfactory. On occasions when only a few copies are required and a negative image will serve the purpose equally as well as a positive, bromide paper may be used in the dark slide instead of the plate, this being much more economical. Although a half-plate camera is very suitable for

this work, any smaller sized plate may be employed by using adapters in the dark slide.

ARRANGEMENT OF APPARATUS: *First Method.*—The camera is fitted with a short focus (10 to 12 cm.) single meniscus photographic lens, and is then firmly fixed to the working bench. The object is placed in position and focussed roughly by altering its distance from the lens, the final focussing being done by adjusting the front or back of camera. By this method magnifications up to about $\times 4$ may be attained.

Second Method.—The arrangements adopted in the first method are used with the addition of a large diameter lens on a separate support between the camera lens and the object. This will enable a magnification of about $\times 8$ to be obtained before the optical qualities of the camera lens are upset sufficiently to produce poor images on the plate.

Third Method.—The general arrangements given above are adopted, but the camera lens is removed and replaced by a microscope objective fitted into a cork. According to the focal length of objective used (76 to 8 mm.) and the extension of camera, magnifications varying from $\times 5$ to $\times 40$ may be obtained without any difficulty in manipulation. It is possible with transparent objects to use an objective of only 4 mm. focal length and attain a magnification of about $\times 75$, but the difficulty of focussing by means of the camera rackwork is great.

Fourth Method.—This method, which allows of magnification up to $\times 400$, requires the use of a microscope and is therefore somewhat outside the scope of this paper. It will be sufficient to mention that the microscope is placed horizontally, and the camera without any lens is placed with its lens mount against the eyepiece. In order to keep the camera fairly rigid, and at the same time allow of its ready removal for observation through the microscope, it is placed upon a pile of paper-covered magazines and has a wooden bar with weights suspended from each end laid across the base-board.

ILLUMINATION OF OBJECTS.—The method to be adopted for illumination depends chiefly upon whether the object is opaque or transparent, and secondly upon the detail required to be shown in the finished print. Transparent objects are usually photographed by transmitted light, and may be illuminated by (a) light passing through direct from the illuminant to the objective; (b) polarised light, by using a polariser and analyser before and behind the objective; and (c) black background obtained by the use of a 4-inch condenser having a central stop to prevent direct light from the illuminant reaching the objective, while the object is illuminated by an annular cone of rays from the periphery of the condenser.

When photographing opaque objects the light must be reflected by the object, which may be illuminated (a) by direct light from illuminant on the same side of the object as the objective; (b) by direct light with the use of a condenser between illuminant and object; (c) a Lieberkuhn reflector fitted to objective.

With objects which are not flat and require to be photographed with sharp detail in various planes, it will be necessary to reduce the aperture of the objective by placing black paper stops at the back which may be held in position by a small screw cap or a circular steel spring.

Since lenses of long focal length require a comparatively large even illumination when dealing with transparent objects, it is advisable to insert one or two pieces of ground glass between the illuminant and object. The increase in exposure necessitated by this addition is shown below:

One piece of ground glass	...	...	...	...	× 1.3
Two pieces of ground glass	...	...	...	...	× 1.5

EXPOSURE.—The determination of the correct exposure required is a difficult problem in photomicrography, and up to the present no exposure meter has been produced which is of any use for the purpose. The exposure depends upon a number of factors, among them being—the colour of the object, the method of mounting illumination (transmitted or reflected), actinic power of illuminant, aperture of lens used, magnification, speed of plate, etc.

By keeping a full record of the details of each exposure one soon acquires a series which is of great value in the determination of any exposure required for a new object. My own record contains spaces for twenty-one particulars in addition to a column for "remarks," but it is rarely necessary to fill up more than half of these.

The following table is an abbreviated form of my exposure record, and the figures given may be a guide to anyone commencing this work for the first time:

Object.	Mounting.	Magn.	Method.	Illumination.	Illuminant.	Objective.	Stop.	Plate.	Exposure.
Coralline pebble	Dry	2	1	Direct, reflected	Incandescent gas	111 mm.	F. 8	Imp. Ord.	2 min.
Brome grass ...	"	3	1	Dark back-ground	Incandescent gas		F. 22	Imp. N.F.	5 min.
Woven linen ...	"	9	2	Direct, reflected.	Magnesium	111+240 mm.	F. 16	" "	12 in Mg.
Ink and pencil marks	Xylol	11	3	Direct, transmitted.	$\frac{1}{2}$ watt, 50 c.p.	64 mm.	4 mm.	Imp. S.R.	6 in Mg.
Woven horsehair	Balsam	37	3	Polarised	Magnesium			Thames	30 in Mg.
Globigerinia ...	Dry	19	3	Lieberkuhn	$\frac{1}{2}$ watt, 50 c.p.	22 mm.	1.5 mm.	Imp. Ord.	10 min.
Lions on half-crown		10	3	Direct, reflected			4 mm.	Imp. S.R.	
Mercury globules from Reinsch test 0.0001 mg.		320		Direct, transmitted		4 mm. + eye-piece × 5	5 mm. in Abbé condenser		30 min.

THE STAG BREWERY,
PIMLICO.

DISCUSSION.

Mr. W. F. REID remarked that if all schools taught their students to make the best of such simple means as illustrated by Mr. Ward, it would be of great help. He had fitted up several kinds of apparatus, using a vertical camera, the great advantage being that it was less subject to vibration.

With regard to illumination, the use of acetylene was to be recommended if a proper generator were available, as it worked very well. This method was adopted by the Italians in warfare. A great deal also depended upon the kind of light that was used, as it could be split up, and thus different results obtained. One of the

difficulties in photographing with invisible rays was the correct focusing of the apparatus.

In the case of photographing insects, different results were obtained with ultra-violet rays with the visible lights cut off, and an excellent photograph of a portion of the insect could be obtained straight away, without mounting, with the reflected light cut off.

Mr. GILBARD thought much better results in colour work could be obtained in different colour screens—that is, yellow, green and blue—were used. The latter especially was to be recommended, as this would sometimes bring out detail where yellow failed to do so.



NOTE ON THE SOLUBILITIES OF THEOBROMINE.

By RAYMOND V. WADSWORTH.

(*Read at the Meeting, March 3, 1920.*)

IN examining the properties of a sample of pure theobromine it was found that the figures obtained for the solubilities were, in certain cases, notably lower than those published. The solubilities to be found in the literature on the subject vary so much amongst themselves that a revision is necessary. With this aim in view I have redetermined the solubility in most of the commoner solvents. The original error seems to have been due chiefly to Mitscherlich, who, working with impure theobromine, obtained high solubilities, which have been copied by many authorities. The impurity in Mitscherlich's alkaloid seems to have been caffein, for his high figures correspond with high solubilities for caffein. His figures are as follows :

	At 15.5° C.	At Boiling-point of Liquid.
	Per Cent.	Per Cent.
Chloroform	—	1.00
Ether... ..	0.006	—
Water	0.160	1.81

Theobromine is only soluble with difficulty in most liquids, and precipitates very slowly. Consequently high figures for solubilities at low temperatures can be easily obtained if care is not taken to allow a considerable time for precipitation after the liquid has been heated. Also low results will be obtained if solution is performed in the cold, unless due precautions are taken. The following is the table of solubilities worked out. All the figures were estimated on the same sample, which I prepared and purified :

Solvent.	Solubility of Theobromine in Grms. per 100 c.c. at—	
	15.5° C.	Boiling-point of Liquid.
	Per Cent.	Per Cent.
Ether (0.720)	0.003	0.003
Benzene	0.005	0.010
Trichlorethylene	0.020	0.030
Carbon tetrachloride	0.020	0.040
Chloroform	0.060	0.070
Alcohol (absolute)	—	0.100
Alcohol (90 per cent.)	—	0.390
Water	0.060	0.700
Tetrachlorethane	0.090	0.870
Aniline	0.650	8.000
Ether petroleum	Insoluble.	Insoluble.

The following are the chief examples of differences between my results and those of other chemists :

	Per Cent.	
<i>Trichlorethylene</i> —		
At 15.5° C.	Up to 7 0.02	Gowing-Scopes (ANALYST, 1914, 39 , 12). Wadsworth.
<i>Chloroform</i> —		
At boiling-point	1.00 0.07	B. P. C., Allen, Thorpe, Mitscherlich. Wadsworth.
<i>Alcohol</i> (90 per cent.)—		
At boiling-point	2.13 0.39	B. P. C., Blyth, Thorpe. Wadsworth.
<i>Water</i> —		
At boiling-point	1.81 0.70	B. P. C., Blyth, Mitscherlich. Wadsworth.

The work was carried out in Messrs. Cadbury Bros. Research Laboratory, under Mr. A. W. Knapp, and I wish to thank the firm for their permission to publish the results.



ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Formation of β -Methylumbelliferone as a Reaction of Acetylacetic Acids and its Esters. V. Arreguine and E. D. Garcia. (*Ann. Chim. anal. appl.*, 1920, 2, 36-41.)—The condensation of the ethyl ester of acetylacetic acid with resorcinol in presence of hydrochloric acid yields β -methylumbelliferone, which is characterised by giving an intense blue fluorescence in extremely dilute alkaline solutions. From 0.1 to 0.2 gm. of resorcinol is mixed with 2 to 3 c.c. of the solution of the ethyl ester in concentrated hydrochloric acid, and the liquid heated for a few minutes up to boiling-point, then cooled, diluted with a little water, and rendered alkaline with ammonia solution. The test is capable of detecting 0.00002 gm. of acetylacetic acid or its derivatives in 1 c.c. of liquid, and the reaction is not given by acetone or β -hydroxybutyric acid. In applying the test to urine 50 c.c. of the sample are acidified with 2 or 3 drops of hydrochloric acid, and shaken with 5 c.c. of carbon tetrachloride or chloroform. The extraction is repeated with 3 c.c. of carbon tetrachloride, and the united extracts are evaporated to 2 to 3 c.c., and heated with 0.1 gm. of resorcinol and 2 c.c. of hydrochloric acid until the solution has evaporated. The liquid is then cooled, diluted, and rendered faintly alkaline with ammonia solution.
C. A. M.

Rapid Method of Estimating Water in Crude Camphor. K. W. Lane and O. F. Lubatti. (*J. Soc. Chem. Ind.*, 1920, 39, 50T.)—Five grms. of the crude sample in coarse granular fragments are placed in a centrifuge tube having a barrel of 25 mm. and a narrow tube of 4 mm. internal diameter, the latter being sealed at the lower end and graduated. Resting on the coned junction between the narrow and wide parts of the tube is a small funnel of nickel-wire gauze, on which is placed a piece of cotton-wool to act as a filter. Thirty c.c. of benzene, previously saturated with water at laboratory temperature and centrifuged before separating the water in a separating funnel, is then introduced into the tube containing the camphor, which it dissolves, and the whole is spun for a couple of minutes. The water in the sample is found in the narrow tube, any insoluble impurities being retained by the cotton-wool. The percentage of water is read off direct, and any particles of water which may have been retained mechanically by the wool or on the walls of the tube are disturbed by carefully stirring with a glass rod and a second reading taken. If a sample is very pure the gauze filter may be dispensed with.
H. F. E. H.

Cryscopic Method for the Determination of Cane Sugar. H. H. Dixon and T. G. Mason. (*Scient. Proc. Roy. Dublin Soc.*, 1920, 16, No. 1, 1-8; through *J. Soc. Chem. Ind.*, 1920, 39, 201A.)—A method for the determination of cane sugar

in small volumes of liquid is based on the fact that after inversion the depression of freezing-point of a cane-sugar solution is approximately doubled. Invertase is added to the sap or other liquid in the cold and the freezing-point determined, after which the mixture is incubated at 30° C. for forty-eight hours and a second observation made. Advantages of the proposed method are that no treatment to eliminate proteins and other colloids is necessary, and that only a small volume of liquid—viz., about 2.5 c.c.—is required. Readings of temperature may be obtained with an error of $\pm 0.003^\circ$, and 0.01° corresponds to 0.2 per cent. of cane sugar.

Occurrence of Melecitose in Honey. C. S. Hudson and S. F. Sherwood. (*J. Amer. Chem. Soc.*, 1920, **42**, 116-125.)—It has long been known that bees collect manna during droughts when floral nectar is not abundant. In some mannas the rare trisaccharide melecitose has been found, and one sample from the Douglas fir of British Columbia contained as much as 70 per cent. of this sugar. It appears, therefore, likely that some honeys should be found to contain melecitose, and this has actually proved to be the case. A sample of comb honey of the honey-dew type coming from Port Royal, Pennsylvania, crystallised solidly in nearly all the cells, the crystals proving to be pure melecitose, while two other samples of honey-dew honey from Maryland also contained melecitose crystals. The bees were found to collect a sweet fluid deposited on the young twigs of the Virginia pine (*Pinus virginiana*) by a soft scale insect or aphid, and this fluid itself contains melecitose. Since melecitose has already been identified in the manna of the European larch, the Douglas fir, and the scrub pine, it may probably be present in the sap of most coniferous trees, it being assumed that the insects producing these mannas and exudations do not themselves synthesise the carbohydrate, although this remains to be proved. In the analysis of honeys suspected to contain melecitose, the cane sugar present, when estimated by the Clerget process, must be inverted by means of invertase and not acid, since invertase is without action on the trisaccharide, while acid hydrolyses it. In the future, when a honey is found by the analyst to show a large increase in reducing sugars after acid inversion, but a much smaller increase when using invertase, the presence of melecitose should be suspected. Again, no honey should be condemned as being adulterated with cane sugar on the sole basis of an increase in reducing sugars by acid hydrolysis, since the presence of the trisaccharide can equally well cause such an increase (see Hudson, *J. Ind. and Eng. Chem.*, 1910, **2**, 143-6). Melecitose, after recrystallisation from hot aqueous alcohol and drying at 70° C., shows an $[\alpha]_D^{20} + 88.2$ and a melting-point of 148° C. After hydrolysis, 100 grms. of the sugar has the same reducing power as 69 grms. of d-glucose (*J. Amer. Chem. Soc.*, 1918, **40**, 1456-60). From these data the percentage of melecitose in honey may be calculated. H. F. E. H.

Rapid Method of Estimating Lead in Cassia Oil. O. F. Lubatti. (*J. Soc. Chem. Ind.*, 1920, **39**, 35-36r.)—Cassia oil exported from China is contaminated with lead derived from the leaden vessels in which it is sold. For the rapid colorimetric estimation of this lead by means of ammonium sulphide 5 c.c. of the oil (or 2.5 c.c. if a preliminary test has indicated the presence of more than

0.025 per cent. of lead) are diluted to about 23 c.c. with 90 per cent. alcohol in a Nessler cylinder of narrow diameter (2.5 cm.). The same amount of cassia oil free from lead is diluted in the same way in a second tube, 1 c.c. of ammonium sulphide solution added to each liquid, and a standard solution of lead in 90 per cent. alcohol (1 c.c. = 0.0001 gm. Pb) is added to the blank until it matches the brown coloration of the sample under examination. The liquid is stirred three times after each addition of lead solution. The results thus obtained are slightly high, the average excess being 0.00018 gm. The amount of lead usually present in commercial samples ranges from 0.04 to 0.06 per cent. In a test experiment, in which a pure oil was left in contact with bright lead in closed tubes, which were shaken at intervals, the maximum absorption of lead was 0.074 per cent., and was reached after one month.

C. A. M.

Factors affecting the Oxygen Absorption of Linseed Oil. A. de Waele. (*J. Soc. Chem. Ind.*, 1920, **39**, 48-50T.)—Since linseed oil on exposure to air not only takes up oxygen, but loses some as volatile products, it is to be expected that equilibrium conditions of the oxidised product with the surrounding atmosphere will, to some extent, be determined by the actual proportion of decomposition products present in the latter. A further factor which has to be taken into account is the probable formation of peroxides stable as such in the absence of water, decomposition taking place in the presence of water vapour. A series of determinations were carried out and described by the author, in which variation in weight on exposure under conditions of light and darkness were compared with the figures recorded for variation in atmospheric conditions, results being shown on curves for the differently treated oils. Circular filter papers 11 cm. in diameter were spotted with raw and variously treated linseed oils in such a way as to secure the minimum thickness of distribution by allowing the maximum of spreading to take place. The papers were then suspended by glass hooks and exposed to the air. It is to be noted, however, that the first series received exposure to light only for about eight out of the twenty-four hours, as the experiments were carried out in mid-winter. Weighings were performed every twenty-four hours, and the variations in weight returned as percentages on the weights of oil spotted on the papers. In the curves representing siccatised oils it should be noted that the drier was introduced in such a way as to ensure the minimum of polymerisation and oxidation. The results tend to show that the presence and nature of the drier does not affect the value of the periodic variation in weight after the first period of ten days in the presence of light, while the accelerating effect of daylight is clearly shown. It would also appear that the inverse reading of the pressure of water vapour in the atmosphere has for its main effect the influencing of the "light" curves. Variation in the "darkness" curves seems to be less dependent on the effect of the pressure of water vapour than on that of the relative humidity, while the relative stability of polymerised oil is clearly shown. The author is of the opinion that the liberation of free fatty acid on oxidation is improbable, the high acid values observed in oxidised oils being more probably due to groupings which under suitable conditions of hydrolysis, such as in neutralisation, would function as carboxyl or hydroxyl groups. Such hydroxyl groups attached to

ring nuclei formed by inner polymerisation subsequent to oxidation would function in the same way as phenolic hydroxyl groups, the existence of which are strongly indicated by the high acetyl values of oxidised oils. The samples used for the experiments were raw linseed oil (no drier); raw linseed oil containing 0.1 per cent. Mn, raw linseed oil containing 0.5 per cent. Pb, raw linseed oil containing 0.1 per cent. Pb and 0.0023 per cent. Mn, and, finally, polymerised linseed oil free from oxidised oil or drier.

H. F. E. H.

Hours of Labour as affecting Composition of Milk. G. C. Jones. (*Dairyman*, 1920, 42, 232.)—The author publishes the following table showing the average composition of milk passing through his hands during the ten years ending with 1918. They agree within about one in the second decimal place with those published in the *ANALYST* by Richmond over a period of ten years :

COMPOSITION OF MILK.

	Morning.		Afternoon.	
	Fat.	Non-fatty solids.	Fat.	Non-fatty solids.
January	3.62	8.95	3.91	8.95
February	3.54	8.95	3.84	8.94
March	3.49	8.93	3.80	8.93
April	3.43	8.89	3.76	8.87
May	3.31	8.92	3.76	8.91
June	3.26	8.91	3.74	8.87
July	3.40	8.81	3.80	8.75
August	3.49	8.79	3.94	8.74
September	3.61	8.86	4.05	8.84
October	3.71	8.93	4.11	8.92
November	3.82	8.96	4.14	8.95
December	3.77	8.94	4.02	8.94

The purpose of the paper is to show that recent alterations in the hours of agricultural labour tend to exaggerate the difference in composition of morning and afternoon milk, due to the fact that the interval between morning and afternoon milking is shorter, and that between afternoon and morning milking longer. That unequal milking periods exaggerate the difference in composition between morning and afternoon milk has long been known, Monday morning's milk being notoriously poor, due to early milking on Sunday afternoons.

That the shortened hours of agricultural labour would have the effect described was to be anticipated, therefore, and the author states that he has for some months had evidence that they were operating to an appreciable extent, but whilst milk was at its best in mid-winter even morning milk was of fair quality. In February, however, the author's morning samples averaged only 3.32 per cent. of fat, or 0.22 per cent.

below the decennial average; his afternoon ones 4.06 per cent., or 0.22 per cent. above the decennial average. If these results prove typical, this would appear to demonstrate that the average composition of milk has undergone no change, but that morning milk is poorer than formerly.

Carbonic Acid and Carbonates in Cow's Milk. L. L. Van Slyke and J. C. Baker. (*J. Biol. Chem.*, 1919, 40, 335-344; through *J. Soc. Chem. Ind.*, 1920, 39, 130A.)—The carbon dioxide of milk may be estimated by the process originally proposed for the estimation of carbon dioxide in blood plasma (Van Slyke, *J. Soc. Chem. Ind.*, 1917, 36, 944). Milk is drawn from the cow's udder into a 100 c.c. cylinder so as to fill the cylinder from the bottom upward, thus avoiding mixture with air or loss of carbon dioxide. Two c.c. of this milk is transferred with precautions against loss of carbon dioxide into the Van Slyke carbon dioxide apparatus (*loc. cit.*) and treated with a 20 per cent. solution of lactic acid. This is found a more satisfactory method of liberating the carbon dioxide than by employing sulphuric acid, which clots the milk into large lumps. The carbon dioxide content of milk varies over a wide range; a normal value is regarded as about 10 per cent. by volume. The degree of acidity tends to decrease with an increase of carbon dioxide. The carbon dioxide may be completely removed by exhaustion from a thin film of milk which is kept in motion. After pasteurisation the carbon dioxide content of the milk falls, but the hydrogen in concentration remains the same. The suggestion is advanced that the carbon dioxide content of milk might afford a means of distinguishing heated from normal milk. Probably the carbon dioxide of milk is present as a mixture of 1 part of carbonic acid to 2 of sodium bicarbonate.

Conditions causing Variation in Reaction of Freshly Drawn Milk. L. L. Van Slyke and J. C. Baker. (*J. Biol. Chem.*, 1919, 40, 345-355; through *J. Soc. Chem. Ind.*, 1920, 39, 130A.)—In over 300 samples of fresh milk the value of PH varied from 6.50 to 7.20, being under 6.76 in 80 per cent. of the samples. In the case of twenty cows it was found that the reaction of the milk from different quarters of the udder varied greatly, but in most cases the variations were not large. In general a decrease of acidity is associated with a marked tendency towards a decrease in specific gravity, and in percentage of fat, total solids, solids-not-fat, caseinogen, and lactose, but an increase in proteins other than caseinogen and in ash and chlorine. These changes are such as would be expected if blood serum or lymph were added to normal fresh milk. Abnormal conditions of the udder might cause such additions. Examination of milks of low acidity, PH above 6.80, indicates that the reaction is accompanied by the presence of large numbers of leucocytes, though the reaction in such cases may be neutralised by the presence of large numbers of acid-producing streptococci.

Detection of Abnormal Milk based on Hydrogen Ion Concentration. J. C. Baker and L. L. Van Slyke. (*J. Biol. Chem.*, 1919, 40, 357-371; through *J. Soc. Chem. Ind.*, 1920, 39, 130A.)—One drop of a saturated solution of dibromo-o-cresol-sulphon-phthalein (bromo-cresol purple) in water is added to 3 c.c. of milk

and the colour observed. Normal fresh milk gives a greyish-blue colour. The production of a darker or lighter colour seems to awaken suspicion in regard to the normal character of the milk. The colour is made lighter by acids and formaldehyde, and by heating above the usual point of pasteurisation. The colour is of deeper blue in the case of milk from diseased udders, watered milk, skimmed milk, and milk containing alkaline salts. The method has been applied to 570 samples of market milk and has given trustworthy results. Colour standards can be prepared.

Determination of keeping Quality of Milk. J. C. Baker and L. L. Van Slyke. (*J. Biol. Chem.*, 1919, **40**, 373-382; through *J. Soc. Chem. Ind.*, 1920, **39**, 130A).—Bromo-cresol purple (see preceding abstract) can be used to measure approximately and relatively the keeping quality of milk. The test is applied as given in the preceding paper, with the modification that the pipettes and tubes employed must be sterilised before use, and that the samples under test must be incubated for a given time at 18° to 20° C. The milk is examined for change of colour at twenty-four hour intervals. The main factor shown by this test as related to keeping quality is production of acid, but additional factors to be observed are coagulation of caseinogen, digestion of caseinogen, production of alkali and gas, and development of abnormal odour or taste. In the development of acidity four stages of progress are distinguishable through change of colour. In comparing this test with the bacterial count, it is found that in general large numbers of bacteria and increase of acidity are in fair correlation.

Composition of Hubbard Squash Seed Oil. W. F. Baughman and G. S. Jamieson. (*J. Amer. Chem. Soc.*, 1920, **42**, 152-157).—Hubbard squash (*Cucurbita maxima*) belongs to the pumpkin and watermelon family; the seeds yield about 36 per cent. of oil, having the following physical and chemical properties:

Sp. gr. at 25°/25° C. ...	...	...	...	...	0.9179
$[n]_D^{25^\circ}$...	...	...	...	...	1.4714
Acid value ...	...	...	...	...	0.50
Saponification value ...	...	...	...	...	191.5
Iodine value (Hanùs) ...	...	...	...	...	121.0
Reichert-Meissl value ...	...	...	...	...	0.37
Polenske value ...	...	...	...	...	0.39
Acetyl value ...	...	...	...	...	27.8
Unsaponifiable matter ...	...	...	...	...	1.06 per cent.
Soluble acids ...	...	...	...	...	0.33
Insoluble acids ...	...	...	...	...	94.66 " "
Unsaturated acids ...	...	...	...	...	76.45 " "
Saturated acids ...	...	...	...	...	18.37 " "

The amounts of the different fatty acids present were as follows, calculated as percentages of the oil: Palmitic acid, 12.12; stearic acid, 5.86; arachidic acid, 0.02; oleic acid, 34.09; linolic acid, 43.34.

W. P. S.

Okra Seed Oil. G. S. Jamieson and W. F. Baughman. (*J. Amer. Chem. Soc.*, 1920, **42**, 166-170.)—A sample of okra seed (*Abelmoschus esculentus*) obtained from Avery Island, La., yielded 15.60 per cent. of greenish yellow oil, having the following physical and chemical properties :

Sp. gr. at 25°/25° C. ...	0.9172
$[n]_D^{25^\circ C}$...	1.4702
Acid value ...	1.42
Saponification value ...	195.2
Iodine value (Hanus) ...	95.2
Reichert-Meissl value ...	0.26
Polenske value ...	0.23
Acetyl value ...	21.4
Unsaponifiable matter ...	0.37 per cent.
Soluble acids ...	0.14 " "
Insoluble acids ...	96.2 " "
Unsaturated acids ...	67.33 " "
Saturated acids ...	29.22 " "

The quantities of the different fatty acids, calculated as percentages of the original oil, were as follows: Palmitic acid, 25.82; stearic acid, 2.62; arachidic acid, 0.05; oleic acid, 41.86; linolic acid, 25.47. Specimens of oil obtained from three other samples of the seeds yielded similar results. The oil gives a coloration with Halphen's reagent.

W. P. S.

Estimation of Small Quantities of Nitrous Acid, especially in Salt Meat. F. Auerbach and G. Riess. (*Arbeiten a. d. Reichsgesundheitsamte*, 1919, **51**, 532; through *Chem. Zeit. Übersicht*, 1920, **44**, 17.)—The quantitative colorimetric estimation of small quantities of nitrites with *m*-phenylenediamine is most successful in presence of sodium acetate and acetic acid in known concentration. For the estimation of nitrites in salted meat, the carefully prepared extract must be freed from dissolved albumen and coloured substances by treatment with a colloidal solution of ferric hydroxide. The clear filtrate contains 95 per cent. of any nitrite which may have been present in the meat. Meat pickled for many weeks contains only minute traces of nitrite. If more than 15 mg. per 100 grm. should be found, there is ground for suspicion that the meat has been treated with nitrites.

G. C. J.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Testing the Amylolytic Action of the Diastase of *Aspergillus Oryzae* (Taka-Diastase). S. A. Waksman. (*J. Amer. Chem. Soc.*, 1920, **42**, 293-299.)—The various diastatic enzymes differ in their mode of action towards starch, particularly as regards the relative quantities and rates of formation of the intermediate and final products. In this sense it is convenient to differentiate between the amylolytic (liquefying) and the saccharifying powers of the commercial diastatic preparations. The measurement of saccharifying power has formed the subject of many exact researches, but the measurement of amylolytic power leaves much to

be desired in the way of refinement, and this property is the essential one in the valuation of diastatic enzymes applied to the textile industry. The Lintner method for measuring the saccharogenic action should not be used for comparative studies of different enzymes, since the end products are not the same in all cases. Most of the methods hitherto employed for estimating the starch liquefying power depend on the use of iodine for determining the end point, and the indications are somewhat arbitrary. In the method now described the end point taken to indicate the complete destruction of the starch is the change of the opaque starch paste into a clear solution. To facilitate the recognition of this point it is found to be convenient to dye the starch with neutral red. About 50 to 100 grms. of dry potato starch in a large porcelain dish are wetted with 100 c.c. of a 0.5 per cent. solution of neutral red; the starch is allowed to absorb all the colour, and then washed repeatedly with water until the supernatant liquor remains almost clear. The dyed starch is then dried. For the test a 2 per cent. starch paste is made by stirring the coloured starch with a little cold water, gelatinising with boiling water, boiling for ten minutes, and making up to the required volume. The paste is introduced in portions of 10 c.c. each into large test-tubes, which are then placed in a thermostat at 40° C. When the correct temperature is reached, increasing quantities of the diastase solution are added to the tubes, and these are well shaken and replaced in the thermostat. The end of the reaction, indicated by the clearing up of the colour, is best observed by comparing the liquefied and unliquefied tubes, holding the tubes in the light. The times (T) at which the various tubes become clear are recorded in conjunction with the quantities of enzyme solution (E) present, and it is found that $E \times T = \text{a constant } K$. Then if F = enzyme value at 40° C.; D = dilution multiple of the original enzyme solution; t = standard time (30 minutes); E = quantity of diluted enzyme solution used; and T = the corresponding time of liquefaction,

$$F \frac{30 \text{ min.}}{40^\circ} = \frac{D \cdot t}{E \cdot T} \text{ or } \frac{D \cdot t}{K}$$

In this way the results may be checked by taking the average of several tubes containing different amounts of enzyme. The method is particularly suitable for the study of enzymes which have a starch liquefying power rather large in comparison with the saccharifying power, as, for instance, "Taka-diastase." J. F. B.

Guanidine Content of Faeces in Idiopathic Tetany. J. S. Sharpe. (*Biochem. J.*, 1920, 14, 46).—The work of Paton and Findlay (*Quart. J. Exp. Physiol.*, 1917, 10, 3) has made it probable that the condition of tetany may be due to gastro-intestinal disturbances associated with the absorption of guanidine from the intestinal tract. The author has examined the faeces of normal children and of children suffering from tetany, with the result that he finds the average percentage of this base in the four tetany cases was 0.075 per cent. of the moist faeces, whilst in the six normals only faint traces, averaging 0.007 per cent., were detected. The average daily excretion of guanidine by the bowel in children suffering from tetany was 0.018 grm. as dimethyl guanidine. The following method was employed: 30 to 50 grms. of the moist faeces were mixed in a mortar with 100 c.c.

of water, a little chloroform and thymol added, and the whole dialysed with a parchment membrane for three days surrounded by 200 c.c. of water. The dialysate is then removed, and more water added for a further two days, the mixed dialysates being united and evaporated to a syrup. Ammonium, potassium, and excess of sodium were removed with chloroplatinic acid, and the platinum removed with sulphuretted hydrogen, which is evaporated off, and the bases then precipitated with saturated alcoholic picric acid. Dimethylguanidine crystals separate almost immediately as small bunches of yellow needle-shaped crystals, which are very insoluble in water, melt at 230°C ., and contain 25 per cent. of nitrogen. A loss of about 5 per cent. is incurred by this method, as found by adding known amounts of a guanidine salt to normal faeces, and may be allowed for. An advantage of the method is the elimination of all suspended matter without the use of precipitants.

H. F. E. H.

Detection of Indican in Urine and Blood. A. Jolles. (*Med. Klinik*, 1919 15, 814; through *Chem. Zeit. Übersicht*, 1920, 44, 37.)—Instead of converting the indican into indigo according to the methods of Obermayer and Jaffé, the author recommends the following method. The indican of urine consists principally of potassium indoxylsulphate, which when submitted together with thymol to oxidation by means of ferric chloride yields 4-cymol-2 indolindolignone. This substance forms with one molecule of hydrochloric acid a deep violet dye, which is produced when the chloroform extract of the urine or blood is treated with thymol, fuming hydrochloric acid and ferric chloride.

G. C. J.

Analysis of the Swimming Bladder of the Red Mullet. L. and D. Leroux. (*Ann. Chim. anal. Appl.*, 1920, 2, 47-48.)—The swimming bladder of a specimen of red mullet (*Trigla pini*) had the following composition: Water, 41.65; nitrogen, 4.14; fat, 30.86; and mineral matter, 1.05 per cent. The ash contained 22.2 per cent. of phosphoric acid (P_2O_5), 44.7 per cent. of potassium oxide, and 20.8 per cent. of sodium oxide.

C. A. M.

ORGANIC ANALYSIS.

Analysis of Acetone by Messinger's Method. L. F. Goodwin. (*J. Amer. Chem. Soc.*, 1920, 42, 39-45.)—The following procedure, which is practically the standard recommended generally, gives trustworthy and concordant results in the estimation of acetone by this method. A quantity of aqueous acetone solution, containing from 30 to 40 mgrms. of acetone, is added to 50 c.c. of $\frac{N}{1}$ sodium hydroxide solution contained in a stoppered bottle. After five minutes, about 20 per cent. excess of $\frac{N}{10}$ iodine solution is added while the mixture is shaken continuously. The bottle is then stoppered, and, after a further twenty minutes, 25 c.c. of 2N sulphuric acid are added to neutralise the sodium hydroxide, an excess of 0.3 to 0.4 c.c. of the acid is then introduced, and the iodine is titrated with $\frac{N}{20}$ thiosulphate solution. Each c.c. of $\frac{N}{10}$ iodine solution is equivalent to 0.96747 mgrm. of acetone. Methyl and ethyl alcohols, if present, combine with a certain amount of

iodine. Rakshit (ANALYST, 1916, **41**, 246) has shown that a correction of 1.6 c.c. of $\frac{N}{10}$ iodine solution must be made for each c.c. of ethyl alcohol, and the author finds the correction for each c.c. of methyl alcohol is 1 c.c. of $\frac{N}{10}$ iodine solution.

W. P. S.

Micro-Method for the Estimation of Acetone. M. Richter-Quittner. (*Biochem. Zeitsch.*, 1919, **93**, 163-172; through *J. Soc. Chem. Ind.*, 1920, **39**, 206A.)—A micro-method in which 1 to 2 c.c. of urine and 1.5 to 3 c.c. of alkali need only be used. The urine is distilled once with steam in the presence of acetic acid and a second time with dilute sulphuric acid. Blood or plasma need only be distilled once, and instead of the steam, air is passed through the heated flask. The titration of the distilled acetone is carried out with $\frac{N}{100}$ iodine and $\frac{N}{100}$ sodium thiosulphate; 0.1 mgrm. of acetone in 100 c.c. can be estimated with accuracy by this method. The quantity of urine and blood used must not contain less than 0.04 mgrm. of acetone.

S. P. H.

Determination of Amino Acids by Means of the Hydrogen Electrode. E. L. Tague. (*J. Amer. Chem. Soc.*, 1920, **42**, 173-184.)—The colorimetric methods for the estimation of hydrogen ions do not differentiate between the excess of alkali necessary to overcome hydration and the amount of alkali necessary to neutralise the solute. Strictly speaking, neutralisation consists in bringing the concentration of hydrogen ions to 1×10^{-7} grm. per litre, or $P_{H} = 7.00$. Amino acids, however, require the presence of an excess of hydroxyl ions before they will show their maximum acid characteristics, or an excess of hydrogen ions before they will show their maximum basic nature. In the titration of amino acids, even in the presence of formaldehyde, a certain proportion of the added hydroxyl ions are used up in bringing the solvent itself to the hydroxyl ion concentration required for complete neutralisation of the solute. The hydrogen electrode furnishes a means for the accurate determination of this factor. The ionisation equilibria of an amino acid are produced by its functions as an acid, as a base, and as an inner salt, and the inner salt tends to hydrate as the solution approaches the point of neutrality. As soon as the concentration of the hydroxyl ion is sufficient to reduce the concentration of the hydrogen and hydroxyl ions, resulting from the dissociation of the inner salt, to the same value as that of the ions in pure water, the solute has been quantitatively neutralised. The investigation of the amino acids resolves itself into a study of three factors: the behaviour of (1) the solute plus solvent, (2) the solvent alone, (3) the solute alone, the value of the third being calculated from the difference between the other two. An hydroxyl ion concentration of about 2×10^{-2} ($P_{H} = 12.5$) will suppress to a negligible quantity the basic ionisation of the sodium salts of the amino acids and thus make possible a more exact quantitative determination of the diamino acids, as well as of others containing strongly negative groups. Sufficient standard alkali is added to a definite volume of the aqueous solution of the amino acid to give it a P_{H} value of about 12.5. Then to an equal volume of water the same standard alkali solution is added in an amount sufficient to give it the same P_{H} value, care being taken to add sufficient water to the blank solution to make its final volume the same

as that of the amino acid solution containing the alkali. The difference between the c.c. of alkali used in the two liquids adjusted to the same P_H value gives the quantity of alkali required to neutralise the amino acid. The apparatus used comprised a Kohlrausch slide wire bridge, a type B (No. 2,500) Leeds and Northrup galvanometer, a Weston millivoltmeter and multiplier, Edison storage cells and the hydrogen and normal calomel electrodes made according to the directions of Hildebrand. Decinormal solutions of the amino acids and sodium hydroxide were employed and the initial volume of liquid was in all cases made up to 100 c.c. The electrode vessel was protected from the carbon dioxide of the air by a rubber stopper through which the electrodes and the tips of the burettes were inserted. Hydrogen gas was passed through the liquid in the electrode vessel at a constant temperature of 18° C. until equilibrium was attained and the constant potential was noted. This required 30-60 minutes, the vessel being continuously shaken at the rate of 50-60 strokes per minute.

J. F. B.

Reagents from *n*-Butyl Alcohol. R. Adams and C. S. Marvel. (*J. Amer. Chem. Soc.*, 1920, **42**, 310-320.)—*n*-Butyl alcohol is now available in commercial quantities as a product of the fermentation of starch in the manufacture of acetone and a number of organic chemical reagents may be prepared, starting from *n*-butyl alcohol as the raw material. For instance, butyl bromide is obtainable in almost quantitative yields and is easily converted into butyl cyanide. This product by reduction readily gives *n*-amylamine, or by hydrolysis with alkali, or by treatment with sulphuric acid and an alcohol it will give *n*-valeric acid or its esters respectively, compounds which have always been difficult to obtain in the pure state. By the reduction of ethyl *n*-valerate, *n*-amyl alcohol can be produced in good yields. Thus it is possible to obtain directly or indirectly the various *n*-amyl derivatives which have only rarely been available to the organic chemist. Another group of compounds most readily synthesised by means of *n*-butyl alcohol are *n*-caproic acid and its derivatives. Caproic acid is interesting on account of its wide distribution in many fats and oils. Its α -amino derivative, known as nor-leucine, occurs among the hydrolytic products of brain protein. Pure caproic acid can be very readily prepared by condensing *n*-butyl bromide with ethyl malonate to form *n*-butylmalonic ester, then saponifying and decomposing this latter compound. The α -bromocaproic acid is best obtained by the bromination of *n*-butylmalonic acid and subsequent loss of carbon dioxide. The α -amino derivative (nor-leucine) is made directly from the bromocaproic acid. Detailed directions for the preparation of the compounds enumerated above are given in the paper.

J. F. B.

Deterioration of Cotton on Wet Storage. N. Fleming and A. C. Thaysen. (*Biochem. J.*, 1920, **14**, 25-28.)—Exposure to damp renders cotton brittle and short fibred and increases its alkali solubility. Loss amounting to 10 or 15 per cent. is often thus entailed in the shape of exceptional short fibres, the so-called "fly." Bacteria are responsible for this deterioration, which can be prevented if the moisture content of the cotton is reduced below 9 per cent. The present paper deals with the results obtained in the investigation of the bacterial changes and with a method

for the quantitative determination of such deterioration. Direct microscopic examination, even with an oil-immersion lens, was found impracticable owing to the minute size of the attacked fibres, and it was therefore found necessary to make use of Cross and Bevan's viscose process for the preliminary treatment of the material as employed by Balls for the demonstration of the daily layers of growth in the cotton hair-cell wall; so treated the cotton fibre swells to about five times its normal size. From 0.1 to 0.3 grm. of the sample is boiled for a short time in 1 per cent. caustic soda solution, washed in running water, and then dipped in 1 per cent. acetic acid, and again washed. After squeezing out excess water, the sample is treated in a small, wide-mouthed, glass-stoppered bottle with 1.5 c.c. of 15 per cent. caustic soda and 1.5 c.c. of carbon bisulphide. In this mixture the fibres are left for fifteen to forty-five minutes and are then ready for microscopic examination, a drop of water being allowed to diffuse under the cover-glass after mounting the fibres. Photographs are appended showing the appearance of the cotton. The method may be made quantitative as regards detection of admixed damaged fibres by treating as above described and following the average of ten counts from each preparation under the microscope. Mixtures of known composition were thus examined, and extremely close results obtained. Further details of the technique will be published later.

H. F. E. H.

Quantitative Estimation of Traces of Dichloroethyl Sulphide (Mustard Gas) in Air. M. Yablick, G. St. J. Perrott, and N. H. Furman. (*J. Amer. Chem. Soc.*, 1920, **42**, 266-274.)—The method consists in reducing a 1 per cent. solution of selenious acid in diluted sulphuric acid (1 : 1 by volume) by means of the dichloroethyl sulphide vapour to an orange-red suspension of selenium, the solution being heated to about 85° C. to facilitate the reaction. By means of the nephelometer the mustard gas can be detected down to the limit of 0.005 mgrm., and amounts from 0.01 mgrm. to 0.001 mgrm. can be estimated with a fair degree of accuracy. The standard solutions of dichloroethyl sulphide in 1 : 1 sulphuric acid contained 0.0078 and 0.00078 grm. per 100 c.c. respectively. To 5.8 c.c. of the standard solution were added 4.2 c.c. of sulphuric acid 1 : 1, and 10 c.c. of selenious acid solution containing 1.0 grm. of selenium dioxide per 100 c.c. of 1 : 1 sulphuric acid. The mixture was heated at 85° C. for ten minutes, then cooled and examined in the nephelometer. For the absorption of mustard gas from air, the bubbling apparatus was designed to pass the air at the rate of 1 litre per minute. Ten c.c. of the absorbing agent was placed in the bubbler and the air mixture passed through it for ten minutes. The liquid from the bubbler was then made up with sulphuric acid so as to yield 20 c.c. of a 1 per cent. solution of selenious acid in sulphuric acid 1 : 1 by volume. When comparing the efficiencies of various absorption reagents a 1 per cent. of selenious acid in water was first adopted, but it was found that a 1 per cent. solution of selenious acid in 1 : 1 sulphuric acid gave more consistent results as an absorbing medium. The selenious acid solution should not be used until twenty-four hours after making up, but it is then stable for at least two weeks.

J. F. B.

Estimation of Cyanides, Cyanates, and Bromides in the Presence of Each Other. G. Velardi. (*Gazz. Chim. Ital.*, 1919, **49**, 317.)—Cyanides are estimated in an aliquot part of the alkaline solution by the ordinary method of Liebig. Another portion is exactly neutralised with acetic acid, and the total volume of silver nitrate solution required by the bromides, cyanides, and cyanates together is determined by Mohr's method. A third portion is acidified with nitric acid, and the cyanides and bromides estimated by titration with silver nitrate by Volhard's method. Cyanates are not precipitated by silver nitrate from an acid solution. The amount of bromides is then found by difference.

C. A. M.

Estimation of Organic Chlorine or Bromine by the Chromic Acid Method. P. W. Robertson. (*Chem. News*, 1920, **120**, 54.)—The author has previously described a method (*ANALYST*, 1915, **40**, 413) depending on heating the substance with chromic anhydride and concentrated sulphuric acid in a simple apparatus, absorbing the volatile halogen compounds thus produced in a solution of sodium hydroxide containing hydrogen peroxide and then titrating with $\frac{N}{10}$ silver nitrate. The chromic acid may be replaced by potassium dichromate. Whereas perhydrol was formerly recommended because few other commercial brands of hydrogen peroxide were chlorine-free, the author now recommends the use of a cold, freshly prepared solution of sodium peroxide, which is almost invariably halogen-free. Finally, $\frac{N}{20}$ solutions of silver and of thiocyanate are recommended in place of $\frac{N}{10}$ solutions. Trouble has sometimes been found with liquid substances, which are apt to be oxidised with explosive violence. This can be avoided by covering the substance with a rather large amount of dichromate and running in the cooled acid very quickly, cooling the reaction vessel in melting ice.

G. C. J.

Estimation of Fluorine in Organic Substances. E. Paternò. (*Gazz. Chim. Ital.*, 1919, **49**, 371.)—The combustion of organic substances containing fluorine in Berthelot's calorimetric bomb with oxygen under a pressure of 25 atmospheres does not give good results in the presence of water or of alkaline solutions. If, however, the combustion is made in presence of a mixture of potassium iodide and iodate, and the liberated iodine titrated with sodium thiosulphate solution an accurate estimation of the fluorine is possible.

C. A. M.

Estimation of Glycerol in Presence of Trimethyleneglycol. C. A. Rojahn. (*Ber.*, 1919, **52**, 1454; through *Chem. Techn. Übersicht*, 1920, **44**, 4.)—Glycerol obtained by the fermentation process may contain trimethyleneglycol, and the crude liquors obtained in the process are likely to contain it in appreciable amount. In such glycerol and such liquor, therefore, it is useless to attempt to estimate the glycerol by Zeisel's method, because trimethyleneglycol is converted almost quantitatively into di-iodopropane, so that the presence of 1 per cent. of trimethyleneglycol will cause glycerol to be overestimated by 2.42 per cent. The oxidation method may be employed for the analysis of such mixtures, but the most important estimation is that of distillable glycerol.

G. C. J.

Estimation of Paraffins in Commercial Toluene. E. Lewis. (*J. Soc. Chem. Ind.*, 1920, 39, 50r.)—The author refers to the paper by H. G. Evans (ANALYST, 1920, 54), and describes a method in use in many of the T.N.T. factories during war time. The estimation was actually carried out on the mononitrotoluene, and the paraffin content calculated back to the original toluene. One litre of the mononitro compound was submitted to fractional distillation in a three-section Young's evaporator column, collecting that portion which distilled below 160° C. The distillate consisted of a small amount of the mononitro compound, together with the whole of the unnitrated toluene and paraffin. Extraction with 90 per cent. sulphuric acid removes the nitrotoluene, while sulphonation with 20 per cent. oleum and subsequent extraction with 90 per cent. sulphuric acid removes the toluene, the residual paraffins being weighed, and a solubility correction applied. Sulphonation of a large amount of toluene, which is difficult to regulate, is thus avoided, and, if required, the unnitrated toluene may be estimated by weighing before and after sulphonation. The paraffin present in toluene supplied by the Gas, Light, and Coke Co., Beckton, was found to consist almost entirely of isomeric octanes, which, in spite of surviving the initial nitration, were decomposed during the more vigorous final nitration of the toluene into trinitrotoluene.

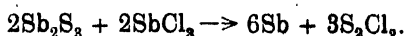
H. F. E. H.

Estimation of Paraffins in Commercial Toluene. H. G. Colman. (*J. Soc. Chem. Ind.*, 1920, 39, 50r.)—The method described above (see preceding abstract), whilst giving a fairly accurate estimation of the true paraffin hydrocarbons (C_nH_{2n+2}) series, in commercial toluene does not give a correct figure for the total percentage of saturated aliphatic hydrocarbons in the original toluene. Since these consist not only of true paraffins, but also to a considerable extent of cyclo-paraffins, these are to a very material extent attacked and removed, both in the original nitration of the toluene, and in the subsequent treatment with 20 per cent. oleum, described by Lewis.

H. F. E. H.

INORGANIC ANALYSIS.

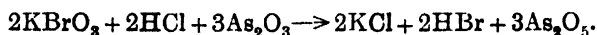
Formation and Nature of Black Antimony Sulphide. F. de Bacho. (*Annali Chim. Applic.*, 1919, 12, 143-152.)—The transformation of red antimony sulphide into the black modification takes place under varying conditions. When it is dried in a current of carbon dioxide or hydrogen sulphide, the change takes place at about 207° to 212° C. The sulphide loses about 1 per cent. in weight (about the same as *in vacuo* over sulphuric acid), and there is no material difference in the loss at 150° C. or 300° C. The presence of 50 per cent. of antimony chloride lowers the temperature of transformation to 115° C. Similarly, in the preparation of the black sulphide by precipitation, the process is promoted by the presence of antimony chloride, whether added as such, or formed by adding hydrochloric acid or ammonium chloride. In this case a reaction takes place between the red antimony sulphide and the antimony chloride, which is probably as follows:



In the formation of the black modification by the dry method, in the absence of antimony chloride there is slight dissociation, with the liberation of metallic antimony, but this is not capable of gravimetric estimation by the ordinary analytical methods. This liberated antimony dissolves in colloidal form as a solid solution in the antimony sulphide, and thus forms the black modification. There is no pronounced difference in the sp. gr. of the two modifications. The sp. gr. gradually rises with the temperature of heating, but there is no sudden rise when the change into the black modification occurs.

C. A. M.

Volumetric Reduction Method with Arsenious Acid. F. de Bacho. (*Annali Chim. Applic.*, 1919, 12, 153-174.)—A new method of estimating oxidising substances is based upon their reduction by means of arsenious acid in the presence of hydrochloric acid, and direct titration of the excess of the reagent by means of potassium bromate—



Methyl orange, which is decolorised by a slight excess of free bromine, is used as indicator. To prevent loss of arsenic chloride by volatilisation the amount of free hydrochloric acid in the solution must not exceed 12·8 per cent. At 15 per cent. the loss was 0·44 per cent., and at 19·4 per cent. it was 48·48 per cent. For the estimation of permanganates the concentration of the acid may vary between 5 and 14 per cent., but a large excess of the arsenious solution must be used. Oxidation is complete in five to ten minutes at the ordinary temperature, and the liquid is then diluted and titrated with $\frac{N}{10}$ potassium bromate solution until a yellow coloration appears. It is then titrated with the arsenious solution until the colour disappears, and finally titrated back with the bromate solution, after the addition of 2 drops of 0·1 per cent. methyl orange solution. In estimating manganese dioxide in pyrolusite by this method 0·1 gm. of the finely powdered sample is treated with 40 c.c. of $\frac{N}{10}$ arsenious acid solution, 15 c.c. of water, and 25 c.c. of concentrated hydrochloric acid, and boiled under a reflux condenser until solution is complete, after which the liquid is reduced and titrated as in the case of permanganates. In the case of barium peroxide the reduction is effected slowly but completely at the ordinary temperature, but much more rapidly in a boiling solution. The concentration of the hydrochloric acid may vary from 6 to 12 per cent. without affecting the results. Chlorates are estimated by treating 15 c.c. of the solution of about $\frac{N}{10}$ strength with 40 c.c. of $\frac{N}{10}$ arsenious acid solution and 25 c.c. of concentrated hydrochloric acid, boiling the liquid for at least fifteen minutes beneath a reflux condenser, cooling it for ten minutes still beneath the condenser, diluting it with 50 to 60 c.c. of water and titrating it with the bromate solution. The active chlorine in bleaching powder or hypochlorites may be estimated in the presence of chlorates by keeping the concentration of the hydrochloric acid at 5 per cent., and effecting the reduction in the cold, whilst by increasing the acid concentration to 10 to 12 per cent., and effecting the reduction at boiling temperature, both hypochlorites and chlorates are estimated. Perchlorates do not interfere with the estimation. The method also gives good results with chromates, including lead chromate.

C. A. M.

Use of Arsenious Acid in the Volumetric Method of Namias. F. de Bacho. (*Annali Chim. Applic.*, 1919, 12, 136-142.)—The volumetric method of Namias (*Gazz. Chim. Ital.*, 1892, 22, 508) consists in treating any given oxidising agent with an excess of arsenious anhydride in the presence of ammonium acetate, effecting the reduction in boiling solution, and titrating the excess of arsenious acid with iodine in the presence of acetic acid. It is shown that this method gives inaccurate and inconcordant results, and that the use of starch paste as an indicator is inapplicable in a boiling solution, no blue coloration being obtained at 60° C. to 70° C. The presence of ammonium chloride, which is formed when the original solution contained hydrochloric acid, has also an influence on the results. The estimation of chlorates and manganese peroxide in a boiling solution containing excess of hydrochloric acid is liable to result in a loss of arsenic as arsenic chloride by volatilisation. Contrary to the assertion of Namias, the reduction of chromic acid by arsenious acid in the presence of hydrochloric acid is not complete in a few minutes at the ordinary temperature, but requires prolonged action. On the other hand, the results thus obtained are too high, and not constant. C. A. M.

Determination of Chlorine with the Nephelometer. A. B. Lamb, P. W. Carleton, and W. B. Meldrum. (*J. Amer. Chem. Soc.*, 1920, 42, 251-259.)—The nephelometric method has been applied to the estimation of toxic "war gases" in air, including mustard gas, which necessitated the use of alcoholic solutions for absorbing the gas. The Kober nephelometer was employed and several details in connection with the manipulation of this instrument, with a view to avoiding appreciable errors, are described. These details were of special importance in the present work, because the extremely minute quantities of chlorine to be determined required the utmost refinement of the method. The standardisation readings were made by adding sufficient alcohol to appropriate quantities of very dilute sodium chloride solution to give a volume of 20 c.c., then adding 10 c.c. of $\frac{N}{10}$ nitric acid, followed by 10 c.c. of $\frac{N}{200}$ silver nitrate, making a total volume of 40 c.c., consisting approximately of 50 per cent. alcohol. The opalescence of a silver chloride suspension changes with time at different rates according to the concentration and other conditions; a sensible time is required for the development of the maximum effect, and this effect then decreases owing to the coalescence of the particles. A standard of constant opalescence may be conveniently prepared by shaking finely-ground kieselguhr with water, allowing it to stand for seventy-two hours and drawing off the supernatant liquor. This suspension had a value approximately equivalent to $N/4.5 \times 10^{-5}$ silver chloride. Using this standard suspension as a constant, the rates of development and decrease of the opalescence of silver chloride were studied at various temperatures and concentrations. As the result of these observations the procedure adopted in the routine analysis was to place the sample, immediately after precipitation, in a water-bath at 40° C. for at least thirty minutes. It was cooled rapidly to room temperature and compared within thirty minutes with standard silver chloride suspensions similarly prepared. Using this method, chlorides could be estimated with an average deviation of about 3 to 4 per cent. over concentrations ranging between 4 and 800×10^{-6} gm.-mol. per litre. Experi-

ments on the effect of light upon the permanence of the opalescence showed that the rate of decrease of opalescence was more rapid when the suspensions were exposed to diffused daylight than when they were kept in the dark, particularly in the more concentrated solutions. Further, it was established that the opalescence of the same quantity of silver chloride in water was about 15 per cent. greater than in 50 per cent. alcohol or 50 per cent. acetic acid. J. F. B.

Copper Flame Test for Halogens in Air. A. B. Lamb, P. W. Carleton, W. S. Hughes, and L. W. Nichols. (*J. Amer. Chem. Soc.*, 1920, **42**, 78-84.)—The apparatus used consists of a roll of oxidised copper gauze, about 10 cm. in length, contained in a quartz tube about 1 cm. in diameter. To make the test, the air to be tested is drawn through the tube while the end of the copper gauze near the inlet is heated to dull redness. After a suitable time, the gauze is removed from the tube and inserted into a non-luminous flame. A green-coloured flame is observed with as little as 0.1 part of chlorine per million of air if the air is passed over the copper at the rate of 1.5 litres per minute for six minutes. Smaller amounts of chlorine may be detected by prolonging the time for the accumulation of the halogen on the gauze. W. P. S.

Sensitive Reaction of Cobalt. J. Bellucci. (*Gazz. Chim. Ital.*, 1919, **49** 294-298.)—The colour reaction given by cobalt with β -nitroso- α -naphthol is capable of detecting 1 mgrm. of the metal in 17 litres of water, and is therefore much more sensitive than the corresponding reaction with α -nitroso- β -naphthol (*ANALYST*, 1918, **43**, 318), the limit of the sensitiveness for which is 1 mgrm. in 1 to 2 litres of water. The coloration ranges from pink to garnet red according to the amount of cobalt, and in some cases an amorphous red precipitate is formed after the liquid has stood for some hours. C. A. M.

Volumetric Estimation of Iron. J. Houben. (*Ber.*, 1919, **52**, 2,072; through *Chem. Zeit. Übersicht*, 1920, **44**, 41.)—In solutions of ferric and ferrous salts containing free mineral acids, the ferric iron and free acid may be estimated by reducing ferric iron by means of hydrogen sulphide and then titrating with $\frac{N}{2}$ alkali hydroxide, making use of ferrous sulphide as indicator. G. C. J.

Estimation of Ferrous Iron. J. M. Kolthoff. (*Pharm. Weekblad*, 1919, **56**, 1,565; through *Chem. Zeit. Übersicht*, 1920, **44**, 41.)—A disadvantage of the iodimetric estimation of ferrous iron is the necessity for working with air-free solutions. The author has worked out a method free from this objection. The ferrous solution contained in a stoppered flask is, after acidification, treated with 25 c.c. $\frac{N}{10}$ potassium bromate, and 10 c.c. 25 per cent. phosphoric acid, then with 5 c.c. of potassium iodide solution (10 per cent.), and two drops of molybdate solution. After five minutes the separated iodine is titrated with $\frac{N}{10}$ thiosulphate. The molybdate is added to accelerate the reaction between bromate and iodide. G. C. J.

Action of Water on Lead. J. F. Liverseege and A. W. Knapp. (*J. Soc. Chem. Ind.*, 1920, **39**, 27-32r.)—Experiments have been made with a slightly alkaline natural water, which, like pure rain water, has the power of eroding lead so that the scales first formed on the metal fall off, leaving a bright surface exposed to further action. This water, from the Caban reservoir, Wales, has an alkalinity, due to bicarbonates, etc., equal to about 0.8 part CaCO_3 per 100,000. Erosion has been found to be due to the action of oxygen in the presence of water, but the variations in the natural proportion of oxygen in water do not appear to influence the amount of erosion. One day is the most suitable period for applying the erosion test, the results of which are influenced by exposure of the water to glass, and by the depth at which the lead is immersed; the greater the depth, the slower being the erosive action. The velocity of erosion falls with the duration of the test. The amount of lead eroded varies directly with the area of the metal exposed, but variations in the volume of water or small changes in the area of water surface have no appreciable effects upon the results. Erosion occurs readily in waters containing no carbon dioxide, but slight variations in the percentages of carbon dioxide in a water do not materially affect the results. The presence of 1 to 2 per cent. of carbon dioxide causes a sudden change from "erosion" to "plumbo-solvency," and the solution of lead takes place more rapidly when oxygen is present in addition to carbon dioxide. The alkalinity of the water is the chief factor, after the presence of oxygen, in determining the amount and the kind of erosion. The addition of small quantities of calcium hydroxide, carbonate and bicarbonate, or of potassium permanganate, decreases erosion, the most effective agent being calcium bicarbonate, 2 parts of which per 100,000 is usually sufficient for the purpose. In the case of calcium hydroxide, the erosion falls to a minimum when about 7 to 10 parts per 100,000 are present. Increasing the calcium hydroxide above this point causes an increase in the erosion, which gradually reaches its original amount. The addition of 5 parts of potassium permanganate per 100,000 was found to form a protective coating, which prevented erosion. Erosion takes place in the absence of bacteria. On the other hand, the bacteria in water are not all destroyed by exposure to the action of lead for six hours.

C. A. M.

Separation and Estimation of Lead and Bismuth. G. Luff. (*Chem. Zeit.*, 1920, **44**, 71.)—The author agrees that in experienced hands the methods of Löwe and Jannasch are the best. In any but experienced hands, however, they lead to very poor results. The following method is said to give good results in the hands of any chemist who will follow the directions given. To the nitric acid solution of lead and bismuth, ammonia is added drop by drop until a faint permanent turbidity results. This is destroyed by the addition of the least possible quantity of nitric acid. Sufficient saturated ammonium nitrite of this solution is added to the lead-bismuth solution, which is diluted to 200 c.c., covered with a clock glass, and heated to boiling. When nitrogen ceases to be evolved, the solution is filtered, and the precipitate of bismuth hydroxide or basic nitrate washed with hot water, and then dissolved in dilute (1:1) nitric acid. The filtrate is evaporated to dryness, and the residue ignited and weighed as Bi_2O_3 . It may contain as much as 5 per cent.

of silica and 5 per cent. of alkali. The former is expelled by treatment with nitric and hydrofluoric acids, after which the alkali can be readily washed out. The filtrate from the bismuth is acidified with acetic acid, treated with potassium dichromate, and the lead chromate is collected in a Gooch crucible, washed with hot water, dried at 120°C ., and weighed.

G. C. J.

Volumetric Estimation of Sulphates by Oxidation of Benzidine Sulphate with Potassium Permanganate. P. L. Hibbard. (*Soil Sci.*, 1919, 8, 61-65; through *J. Soc. Chem. Ind.*, 1920, 39, 188A.)—A modification of the method of Raiziss and Dubin (*J. Biol. Chem.* 1914, 18, 297). The solution for the precipitation of benzidine sulphate should be free from iron, heavy metals, nitrates, phosphates, and organic matter, and should only contain between 0.5 and 4.0 mgrms. " SO_4 ." The liquid (10-25 c.c.) is treated with one drop of phenolphthalein and then with dilute sodium hydroxide till just alkaline. It is then neutralised with $\frac{N}{10}$ hydrochloric acid and 0.1 c.c. added in excess for each 5 c.c. of solution. Keeping the liquid quite cold, 5 c.c. of 0.8 per cent. benzedine hydrochloride solution is added, and after 15-30 minutes the benzidine sulphate is collected in a Gooch crucible, washed with the minimum of cold water, transferred with the asbestos pad to the precipitation flask, and heated with 1 c.c. of 10 per cent. sodium hydroxide solution in a boiling-water bath for a few minutes. Then 10 c.c. of dilute sulphuric acid (1 acid:1 water) is added and hot water to bring the volume to 100 c.c. $\frac{N}{20}$ potassium permanganate is run in until there is 5 c.c. in excess and the solution heated on the water bath for exactly ten minutes. Then 10 c.c. of $\frac{N}{20}$ oxalic acid is added and the titration completed with $\frac{N}{20}$ -permanganate. The factor for the conversion of c.c.s of permanganate solution into mgrms. of " SO_4 " is 0.12. It is advisable to make a blank determination.

Iodimetric Estimation of Sulphides. J. M. Kolthoff. (*Pharm. Weekblad*, 1919, 56, 1413; through *Chem. Zeit. Übersicht*, 1920, 44, 41.)—Contrary to the statement of Treadwell, the author finds that, in the iodimetric estimation of hydrogen sulphide, the resulting sulphur occludes no iodine.

G. C. J.

Separation of Zirconium and Titanium as Phosphates. J. Brown and H. T. Madden. (*J. Amer. Chem. Soc.*, 1920, 42, 36-39.)—A method is described for the gravimetric estimation of titanium in the filtrate from the zirconium phosphate precipitate (cf. ANALYST, 1918, 43, 42). The solution containing zirconium and titanium sulphates is acidified with 1 to 2 per cent. (by volume) of sulphuric acid, and excess of hydrogen peroxide is added, followed by an excess of disodium or diammonium phosphate. After about eighteen hours the zirconium phosphate is collected on a filter, washed with water containing a few drops of sulphuric acid and hydrogen peroxide, dried, fused with sodium carbonate, the melt extracted with water, and the insoluble portion fused with sodium hydrogen sulphate and dissolved in dilute sulphuric acid. The zirconium in this solution is then reprecipitated by the

addition of hydrogen peroxide, and phosphate as described, the precipitate collected, dried, ignited, fused with sodium carbonate, dissolved in sulphuric acid, and the zirconium precipitated with ammonia and weighed as ZrO_2 after ignition. If the zirconium phosphate after the second precipitation is still coloured yellow owing to the presence of a remaining trace of titanium, it must be dissolved and precipitated a third time. The combined filtrates from the zirconium phosphate precipitates are heated to destroy hydrogen peroxide, the volume of the solution adjusted so that it contains 2 per cent. (by volume) of sulphuric acid, an excess of disodium or diammonium phosphate is added, the precipitated titanium phosphate collected after one hour, washed, converted into the dioxide as in the case of the zirconium, and weighed.

W. P. S.

APPARATUS, ETC.

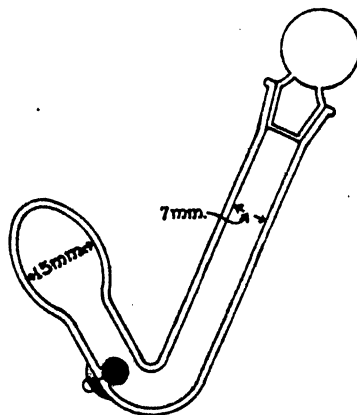
Volumetric Testing of Scientific Glassware at the National Physical Laboratory. J. G. Petavel. (*Report, National Physical Laboratory*, November, 1919.)*—Volumetric glassware has been tested on a small scale at the National Physical Laboratory for the past fifteen years, although its actual manufacture is an industry which was practically non-existent in this country before the war. Latterly, however, the industry has largely developed in this country, and it is desirable that it should remain permanently British. It is quite evident that British-made graduated apparatus should be of reliable accuracy, and many will feel that a reputation for such accuracy could best be guaranteed by an impartial institution such as the metrology department of the National Physical Laboratory. In co-operation with manufacturers and users of scientific glassware this department issued in July, 1918, a pamphlet containing regulations relating to Class A tests—i.e., tests on apparatus required to be of the highest accuracy. A new building has just been completed at the laboratory specially designed for dealing with this class of work on a large scale, and they are now prepared to undertake Class B tests, pending the settlement of a permanent scheme for commercial testing by State or State-approved institutions—a matter which is now under the consideration of the Government. As regards Class B tests, the limits of error assigned are such as all graduated apparatus of good commercial quality should comply with as necessary for the attainment of satisfactory results in ordinary routine analysis. Apparatus graduated under the Class A and Class B limits are etched respectively with the date and monogram of the National Physical Laboratory. Vessels of ordinary type, such as used in volumetric analysis and other customary operations of the chemical or physical laboratory, and measuring vessels for gas analysis, are admitted to the Class A tests. The Class B tests are also available for these types of apparatus, for certain similar types fulfilling somewhat less stringent requirements, and for rain gauges and measuring vessels for milk analysis. The tolerances allowed in a few typical cases are :

* Copies of this report may be obtained gratis from the Director of the National Physical Laboratory, Teddington.

	Class A Limits.	Class B Limits.
2 c.c. Pipette	0.006 c.c.	0.012 c.c.
100 c.c. "	0.05 c.c.	0.08 c.c.
100 c.c. Flask	0.05 c.c.	0.10 c.c.
1,000 c.c. "	0.20 c.c.	0.30 c.c.
50 c.c. Burette	0.04 c.c.	0.07 c.c.

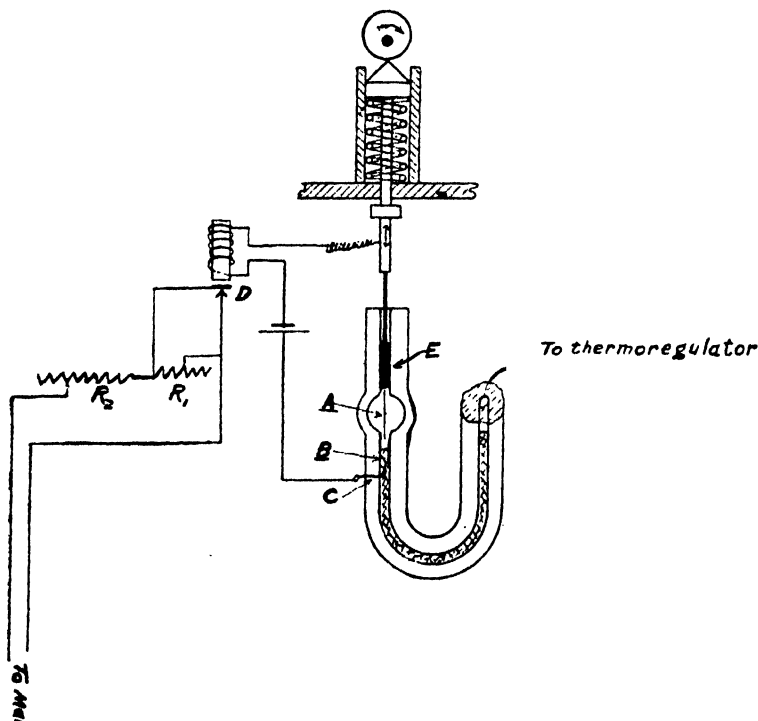
The fees for testing flasks and pipettes range from 6d. to 1s. per instrument. A burette is tested at five points for 2s. 6d. A charge of 6d. extra per instrument is made when certificates are issued. The highest fee charged is 5s. for a gas burette, including certificate. The above particulars as to limits and fees give a brief outline relating to a few special cases only. They are intended to give some idea of the accuracy required and the charges made. A full account of the tolerances allowed, methods of test, details of constructions, test fees, etc., is given in this report which was drawn up in co-operation with both manufacturers and users of volumetric apparatus. Users may send apparatus direct to the laboratory for test, or may specify when placing orders for apparatus that it is to be supplied with the National Physical Laboratory Class A or Class B mark, as required. If a certificate of corrections is desired with a Class A vessel this also should be specified. It is hoped that in the near future dealers will be in a position to supply verified ware from stock. In the event of users of apparatus requiring tests other than those provided for in the pamphlet, the laboratory is always prepared to make special arrangements to meet the particular requirements of any special work. H. F. E. H.

Simple Hydrogen Electrode. C. H. Bailey. (*J. Amer. Chem. Soc.*, 1920, **42**, 45-48.)—An electrode which is simple in construction and operation, has a short interval between the platinised electrode and the potassium chloride solution and in which the platinised electrode is immersed completely, is shown in the figure. The metal electrode consists of a disc of thin gold plate 5 mm. in diameter and is platinised in the usual way. To use the apparatus, the liquid under examination is introduced so as to fill the bulb arm completely, and the open arm to a depth of about 10 mm. above the top of the bend; hydrogen is now admitted through a thin glass tube until the bulb arm is full of gas and the liquid displaced into the open arm. If the latter is not full, more of the liquid is added, the stopper inserted so that excess of liquid is expelled and air excluded, and the apparatus shaken for two minutes. It is then tilted so that enough liquid flows into the bulb arm to just cover the disc electrode, the stopper is removed, and part of the liquid in the open arm is



drawn off. To complete the circuit, the disc electrode is connected with the potentiometer, and a thin siphon tube filled with saturated potassium chloride solution is dipped into the open arm. The end of the siphon in the open arm is drawn out to a fine point, and pieces of filter-paper soaked in potassium chloride solution are forced into the tip by means of a needle; this filter-paper prevents appreciable siphoning action while the readings are taken. The other end of the siphon dips into a vessel filled with saturated potassium chloride solution which in turn is connected with the calomel electrode. The tip of the siphon can be brought very close to the platinised electrode, thus reducing the resistance of the cell. W. P. S.

Gouy Thermoregulator. T. S. Sligh, jun. (*J. Amer. Chem. Soc.*, 1920, **42**, 60-68.)—The working part of the apparatus is shown in the figure; a rise in temperature of the thermoregulator bulb (not shown) causes the meniscus *B* of the mercury in the U-tube to make contact with the platinum wire *A*, whereupon current flows



through the relay circuits opening a contact at *D*, thus introducing an additional resistance *R* in the heating circuit or opening this circuit entirely. The platinum wire *A* is fitted to the guide-plug *E*, and this is given a periodic oscillating motion along the axis of the capillary tube by means of the cam and spring mechanism shown. The range of this motion may be conveniently 0.1 cm., and the period of the

motion one second. Such a regulator will reduce the variations of the bath temperature and the erratic variations due to variations of the mercury surface to a fraction of the values obtained with the usual form of fixed wire thermoregulator. Regulation to within 0.0001°C. may be obtained. In addition, variations of mean bath temperature due to variations in external conditions are reduced to a very considerable extent.

W. P. S.



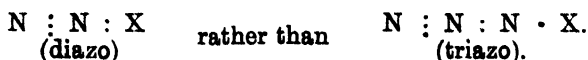
REVIEWS.

THE CHEMISTRY AND TECHNOLOGY OF THE DIAZO-COMPOUNDS. By JOHN CANNELL CAIN. Second Edition. Pp. xi+199. 1920. London: Edward Arnold. Price 12s. 6d. net.

Twelve years have passed since the first edition of this volume was published, and the scope has been somewhat enlarged in the present issue by including many references to the technical literature in order to render it more useful to industrial chemists. In its new form it makes a much wider appeal, and should be acceptable not only to all students of chemistry, but also to those who are occupied with the technology of dye-manufacture.

From the standpoint of systematic nomenclature, adherence to the expressions "diazo-salt," "diazobenzene sulphate," etc., until the concluding chapters is perhaps to be regretted. The word "diazonium" first occurs on page 156, and since it has now been the practice, for many years, to present the subject to students in the light of the diazonium theory, it would increase the value of the work as a class-companion if the introductory chapter, which surveys events from 1858 to 1916, included also a reference to this fundamental principle, and if thereafter the denomination of the respective compounds were harmonised therewith. An added reason for this procedure is offered by the existence of a large class comprising genuine diazo-compounds—namely, those belonging to the aliphatic series; since their inability to form salts distinguishes them sharply from the diazonium compounds, the two names, when systematically used, provide an excellent means of classification.

A minor criticism suggests itself in connection with the section describing the aromatic azoimides. The action of ammonia on diazonium perbromides and of hydrazine on diazonium salts does not involve "replacement of the diazo-group by the azoimino-group" in the sense that this occurs when diazonium salts are mixed with sodium azide, because in the former cases nitrogen is added instead of being eliminated; yet the three reactions are grouped under the heading above quoted. Moreover, the author does not mention the use of the word "triazio" in place of "azoimino," although it is more convenient and more accurately descriptive, since "azoimino" represents literally the arrangement



The maze of conflicting theories regarding the constitution of diazonium salts and of the metallic derivatives from the respective bases, associated more particularly

with the names of Hantzsch and Bamberger, has been carefully threaded, and a useful summary of heterocyclic types capable of undergoing diazotisation has been given. The chapter on aliphatic diazo-compounds, however, is not quite commensurate with the interest and importance of the subject, and might very profitably be extended in a subsequent issue. As one instance only, phenylbenzoyldiazomethane (diazodesoxybenzoin) deserves notice in connection with Schroeter's remarkable transformation of that substance into diphenylketene, since it offers the most convenient method of preparing the latter.

Having modified the title of the book suitably to the wider field which it now covers, the author might consider the desirability of reviewing a few typical analytical methods, such as the use of titanous chloride by Knecht. The subject of diazo-chemistry is one of such vast importance, historical, theoretical, and industrial, that it fully justifies a substantial volume being devoted to itself, and the foregoing remarks are offered solely in the hope that this excellent work, when it appears in the third edition, may be improved still further.

M. O. FORSTER.

POPULAR CHEMICAL DICTIONARY. By C. T. KINGZETT, F.I.C. Demy 8vo.; Pp. vi + 368, with seventy-eight figures in text. 1919. London: Baillière, Tindall and Cox. Price 15s. net.

This book embodies a desire to afford the general reader a true conception of the meaning of words that either are peculiar to chemistry or are used in some special sense in the science. The numbers of articles and phenomena that have been defined without use of a scientific word are surprising. Where this has not been achieved, the meaning of the intruding expression is certain of explanation in its alphabetical position, and the reader has not to waste time before his original enquiry is satisfied.

Usually the definitions are simple, pithy, and happy in expression. Sometimes, however, the words are such as might be spoken by one suddenly asked for information, and the result is long, involved, and far too dependent on commas. The book is well-balanced and comprehensive. Except for the oxide, the ferric salts have been overlooked. The treatment of volumetric analysis is weak, the only indicators mentioned being litmus, turmeric, and phenolphthaleïn. The spectroscope is only applied to inorganic bodies. On the other hand, two monographs on the same subject appear under "India-rubber" and "Rubber."

Actual errors are few, and generally trivial: *Cascara sagrada* is defined as an extract, *Cocculus indicus* is said to be synonymous with "grains of Paradise," blood-heat is given as 36.6° C., acetone is credited with a pungent odour, *Cacao-fat* is said to be used in making soap and candles, basic-slag is given as a source of potash, pumice-stone is described as "molten," and a factor of 0.648 for the conversion of grammes to grains is given.

Misspelt words include cajeput, saccharine (for saccharin), saccharometers, conchinine (probably for quinidine), *Aleuritis moluccana*, *Bacillus butylicus*, propionic acid, glycolic acid, wolfram (for wolframium), and *Mycodermi aceti*.

Though there can be no pus without them, leucocytes are not mentioned in

explaining it. It is exaggeration to say that bacteria "require to be magnified many thousands of times to become visible."

Taraxacum is said to be a diuretic; it is generally used as a laxative. It is difficult to see why liquid paraffin should be "a mild substitute for cod-liver oil." The mercury barometer is described without mentioning the vacuum.

Mr. Kingzett is correct in his scientific phraseology, and usually manages to avoid the weird and barbarous words which chemistry has collected to itself through the centuries. A good part of the text has been carried forward from "Chemistry for Beginners," so have most of the illustrations, the latter generally from blocks lent by dealers in apparatus, with the well-known names in the pictures.

WILLIAM PARTRIDGE.

CHEMICAL CALCULATION TABLES FOR LABORATORY USE. By HORACE L. WELLS. Second Edition, Revised. 43 pp. 1919. New York: John Wiley; London: Chapman and Hall. Price 6s. 6d. net

The present work is a revision and modification of the author's "Tables for Chemical Calculations," published sixteen years ago, and now out of print. A new table for facilitating the calculation of percentage composition of organic compounds has been included, while the five-place logarithmic table is provided with a novel double thumb-index, enabling the reader to turn backwards or forwards from any place in the table to any other reference by a single motion, "since all indices are visible from any part of the table." In the copy before the reviewer this is, unfortunately, not quite true, since the registering of some of the marginal cuts is not sufficiently accurate to ensure visibility of all figures. In other respects the work is well printed, and appears to have been compiled with care and accuracy. The tables included deal with atomic weights (1919), gravimetric factors, formula weights, reduction of gas volumes to 0° and 760 mm., calculation of percentage of nitrogen from gas volumes, barometer corrections for temperature, constants for molecular-weight determinations, weights and measures, and the logarithmic tables above alluded to.

H. F. E. HULTON.

CHEMISTS' MANUAL OF NON-FERROUS ALLOYS. By JAMES R. DOWNIE. Pp. 168. 1920. London: E. and F. N. Spon, Ltd. Price 10s. net.

This book describes the analysis of metals and alloys, and its object, according to the preface, is to present a "reliable guide" to chemists interested in non-ferrous alloys, and "to give in as concise a form as possible information which hitherto has been scattered throughout various textbooks." The author would have been well advised had he given a full list of such textbooks; as it is, the manual does not contain a single reference to literature.

From the point of view of reliability the work can only be pronounced a complete failure. It is impossible within the limited space at the reviewer's disposal to enumerate all its shortcomings; sufficient to give some of the more startling statements found in Chapter IX., headed "Refraction of Stock Metals." For the determination of sulphur in copper (p. 105) and in nickel (p. 118), the author recommends

solution in nitric acid, neutralisation with ammonia, and addition of hydrochloric acid before precipitating with barium chloride. This looks suspiciously like an attempt at converting the nitric into a hydrochloric acid solution. In the determination of phosphorus in copper (p. 105), the nitric acid solution of the metal is evaporated twice with hydrochloric acid, presumably with intent to volatilise arsenic (in its higher state of oxidation), for the chloride solution is next treated with molybdate or magnesia mixture. Arsenic in copper is determined (p. 106) by neutralising the nitrate solution with caustic soda, adding 1 gm. of sodium carbonate, re-dissolving the precipitate, and precipitating the solution thus obtained with magnesia mixture. One would like to know what prevents phosphorus from interfering in this determination. In the analysis of tin (p. 107), antimony is titrated in hydrochloric acid solution with thiosulphate after addition of potassium iodide, in presence of iron, copper, and arsenic, while phosphorus is determined (p. 109) with magnesia mixture in the filtrate from the ferric hydroxide precipitated by ammonia. In the determination of cobalt in nickel bullion (p. 118), the nickel from a 10-grm. sample is first eliminated by precipitation with dimethylglyoxime the precipitate having to be filtered off and thoroughly washed. The diameter of the filter required for this operation is not mentioned.

A list of "typical analyses" of metals and alloys is appended. Here the author has clearly committed a double error of judgment; not only has he estimated the principal constituent direct instead of by difference (for none of the complete analyses sum up to a round 100), but, though such direct determinations are hardly accurate to 0.2 per cent., he has calculated the results to the third decimal place, thus: Al 99.014, Sn 0.063, Fe 0.614, Cu 0.048, Si 0.164, Total 99.904 (p. 159). The author does not appear to be over-anxious as to the correctness of chemical formulæ; cobalt pyrophosphate is $\text{Co}_2\text{P}_2\text{O}_4$ (pp. 44, 52, 151), but the correct cobalt factor is given. Bismuth chromate, with the puzzling bismuth factor 0.62561, is given the formula $\text{BiO}_3(\text{Cr}_2\text{O}_3)_2$ (pp. 57, 151). Sodium uranate is $\text{NaO}_2\text{Ur}_2\text{O}_3$, with a uranium factor of 0.876 (pp. 45, 151).

The preface also states that "the processes are given for the sake of clearness in as direct a statement as possible without regard to literary style." The disregard of style is manifest, with impaired clearness as an inevitable result. Numerous misprints, some serious, occur in the text. The book is intended also for the use of beginners, but a less trustworthy counsellor for such it is difficult to conceive.

W. R. SCHÖLLER.



THE INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

At the 42nd Annual General Meeting of the Institute of Chemistry held at 30, Russell Square on March 1, Sir Robert Robertson, Vice-President, occupied the Chair in the place of Sir Herbert Jackson, the President, who was absent through illness. In moving the adoption of the Report of Council, Sir Robert read the President's address, in which reference was made to the position of professional men

under prevailing economic conditions. Notwithstanding the limited number of appointments carrying reasonably high salaries and the difficulty of rising above the standard of mediocrity, most of the professions were attracting such a high proportion of the educated youth of the country that they were likely to be overcrowded. On the other hand, the Government and the industries of the country were becoming more and more alive to the necessity and the wisdom of making appointments for chemists more attractive to the best qualified. In order to secure better remuneration and conditions of appointment chemists were urged to do all in their power to render themselves prepared for such opportunities as came their way. The Institute was ready to help them to secure suitable appointments, and endeavoured to encourage the employment of the competent. The situation was far more promising than at the time of the Armistice. Over 530 chemists whose names had been on the Appointments Register had now no further need of this assistance.

The Institute had been in communication with several of the Whitley Industrial Councils for industries on which chemistry had a bearing, and although under the constitution of the Whitley Councils no provision was made for the direct representation of chemists, several were acting as employers' representatives, and the Councils were prepared, where occasion arose, to turn to the Institute for assistance. The roll of the Institute was steadily increasing, numbering nearly 3,000 Fellows and Associates, and over 500 Registered Students. The Council were engaged in work of reconstruction—remodelling the By-laws, providing for district representation on the Council, and revising the regulations for admission to membership. They had taken up the question of securing representation of chemistry in the Ministry of Health, with the satisfaction of seeing Sir William Tilden appointed a member of the Council on Medical and Allied Services, and Dr. J. F. Tocher appointed Chemist to the Scottish Board of Health. Attention was being given to the continued production by British manufactures of glass apparatus, chemical reagents, and other laboratory requirements which hitherto were obtained mainly from Germany and Austria.

Jointly with the Institute of Metals, a committee was engaged on questions affecting the status and organisation of chemists and metallurgists with the Navy, Army, and Air Force. Mention was made of the activity of the local sections and of the help rendered by them and the Honorary Corresponding Secretaries Overseas in the work of the Institute.

The Officers and Members of Council for the year 1920-21 were elected as follows:

President: Sir Herbert Jackson, K.B.E., F.R.S.

Vice-Presidents: Horatio Ballantyne, Sir James Johnston Dobbie, LL.D., F.R.S., Ernest Mostyn Hawkins, Gilbert Thomas Morgan, D.Sc., F.R.S., Sir Robert Robertson, K.B.E., F.R.S., George Stubbs, O.B.E.

Hon. Treasurer: Edward William Voelcker, A.R.S.M.

Members of Council: Walter Ernest Adeney, D.Sc., A.R.C.S.I., William Bacon, B.Sc., Edward Charles Cyril Baly, O.B.E., F.R.S., Oscar Lisle Brady, D.Sc., Francis Howard Carr, Alfred Chaston Chapman, Allin Cottrell, M.Sc., Alexander Charles Cumming, O.B.E., D.Sc., John Thomas Dunn, D.Sc., Lewis Eynon, B.Sc.,

Alexander Findlay, M.A., D.Sc., Ph.D., George Watson Gray, Frank William Harbord, C.B.E., A.R.S.M., Charles Alexander Hill, B.Sc., Patrick Henry Kirkaldy, Joseph Henry Lester, M.Sc., William Macnab, Samuel Ernest Melling, Gordon Wickham Monier-Williams, O.B.E., M.C., M.A., Ph.D., Andrew More, A.R.C.S., Frederick Mollwo Perkin, Ph.D., George Henry Perry, M.B.E., B.Sc., A.R.C.S., Benjamin Dawson Porritt, M.Sc., Francis Martin Potter, M.B.E., B.Sc., A.R.C.S., John Rogers, O.B.E., Ernest Woodhouse Smith, D.Sc., William Maurice Gathorne Young.

PROFESSIONAL FEES.

THE Council of the Institute of Chemistry have received many communications relating to the fees for analyses and investigations at present charged by private practitioners.

Having regard to the facts that, in addition to the increased cost of living, practising chemists have to pay higher salaries to their assistants and to bear the enhanced cost of apparatus and materials, the Council are of opinion that Fellows and Associates are justified in making a corresponding increase in their fees for professional services.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

ORDINARY MEETING, APRIL 14, 1920.

HELD at the Chemical Society's Rooms, Burlington House, Mr. Alfred Smetham, President, in the chair.

Certificates were read for the first time in favour of Misses Dorothy Gertrude Hewer, B.Sc. (London), Rita Catherine Hawkins Johnson, and Messrs. Harold Hall, F.I.C., Geoffrey Trelawney Bray, A.I.C., Frank William George King, and John Robert Stubbs, M.Sc. (Victoria), F.I.C.

A Certificate was read for the second time in favour of Mr. Laurence Harry Mills, B.Sc. (Birmingham), A.I.C.

The following were elected Members of the Society : Messrs. John Carmichael, F.I.C., Herbert John Evans, B.Sc., F.I.C., Cornelius Durham Garbutt, Douglas Henville, F.I.C., Maurice S. Hutchinson, B.Sc., Benjamin Richards James, F.I.C., Geoffrey Martin, D.Sc., Ph.D., F.I.C., Robert Leitch Morris, F.I.C., and James Wood, M.A., B.Sc., F.I.C.

The following papers were read : "The Turbidity Temperature of Fats, Oils, and Fatty Acids : Part I.," by Albert E. Parkes, F.I.C. ; "The Interpretation of Milk Records," by G. W. Monier-Williams, M.A., Ph.D., F.I.C. ; "The Loss of Free Ammonia from Drinking Water Samples," by A. F. Joseph, D.Sc., F.I.C., and G. A. Freak, B.Sc., F.I.C. ; and "A Decanting and Filter Washing Machine," by Eric Sinkinson, A.I.C.

A NEW METHOD FOR THE ESTIMATION OF METHYL ALCOHOL.

By S. B. SCHRYVER AND CYRIL CHRISTIAN WOOD.

A. Estimation of Methyl Alcohol in Water.

SOME years ago, one of the authors described a method for the detection of formaldehyde, which could be applied to the quantitative estimation of very small amounts of this substance (*Proc. Roy. Soc.*, 1910, B. 82, 226, and reports of Inspector of Foods, Local Government Board, 1909, N. S. No. 12, also *ANALYST*, 1909, 34, 470). The method in question consisted in treating the solution containing the formaldehyde with 1 per cent. solution of phenylhydrazine hydrochloride, then adding potassium ferriyanide, and afterwards concentrated hydrochloric acid, when, in the presence of the aldehyde, a brilliant fuchsine-like colour was produced, which could be detected even when the concentration of formaldehyde did not exceed 1 in 2,000,000. This reaction forms the basis of the method for estimating methyl alcohol described in the present communication.

Several reagents are capable of oxidising methyl alcohol to formaldehyde, but most of these interfere with the application of the test described above. This was not found, however, to be the case when ammonium persulphate was used. If an excess of the persulphate is added, the formaldehyde is oxidised further to carbon dioxide and water. By ascertaining the amount of persulphate that must be added to a given solution of methyl alcohol in water, so that after completion of the reactions under certain standard conditions of experiment formaldehyde just fails to be detectable by the above-mentioned reaction, the percentage of the alcohol present in the water can be estimated with a considerable degree of accuracy, even when the amounts present are as low as 0.0005 per cent.

When ammonium persulphate is heated with a solution of methyl alcohol three reactions can take place simultaneously—viz., the decomposition of the persulphate itself, the oxidation of methyl alcohol to formaldehyde, and the oxidation of the aldehyde. It is, therefore, necessary to specify accurately the conditions under which the estimation should be carried out, as is the case in other oxidation methods, such as that of Fehling's, where several oxidation processes take place at the same time.

The method of carrying out the estimation is as follows: Samples, each of 5 c.c. of the methyl alcohol solution in water are introduced into a series of test tubes (6 inches by $\frac{3}{4}$ inch), and mixed with 5 c.c. of ammonium persulphate solution of varying concentrations. The test-tubes containing these mixtures are then inserted into a water bath containing boiling water, in which they are kept immersed for 10 minutes. At the end of this period 1 c.c. of the mixture is pipetted out from each tube and introduced into a series of smaller test-tubes, each of which contains 1 c.c. of a 1 per cent. solution of phenylhydrazine hydrochloride; to these mixtures are then added 1 c.c. of a 2.5 per cent. solution of potassium ferriyanide and about 3 c.c. of concentrated hydrochloric acid. It will be found

from this preliminary series of experiments that the concentration of methyl alcohol will lie between two limits—viz., a higher limit corresponding to a concentration of the persulphate which is sufficient to oxidise completely the formaldehyde formed in the reaction, and a lower limit in which the concentration is insufficient, and the mixture of persulphate and methyl alcohol solution still gives a positive reaction for formaldehyde after heating for ten minutes. A second series of experiments can then

TABLE I.

Per Cent. Persulphate in 5 c.c. used for Oxidation.	Per Cent. of Solution of MeOH used.	C.c. of this Solution in 10 c.c. Mixture.	Formaldehyde Reaction.	Concentration of MeOH in 5 c.c. Aqueous Solution.
5.0	1.0	1.45	+	0.29
5.0	1.0	1.40	0	0.28
4.0	0.5	2.4	+	0.24
4.0	0.5	2.3	0	0.23
3.0	0.25	3.45	+	0.1725
3.0	0.25	3.40	0	0.170
2.0	0.25	2.3	+	0.115
2.0	0.25	2.25	0	0.1125
1.0	0.1	2.8	+	0.056
1.0	0.1	2.7	0	0.054
0.5	0.1	1.5	+	0.030
0.5	0.1	1.4	0	0.028
0.25	0.05	1.5	+	0.015
0.25	0.05	1.4	0	0.014
0.10	0.025	1.0	+	0.0050
0.10	0.025	0.9	0	0.0045
0.05	0.010	1.3	+	0.0026
0.05	0.010	1.2	0	0.0024
0.025	0.005	1.3	+	0.0013
0.025	0.005	1.2	0	0.0012
0.010	0.001	2.5	+	0.00050
0.010	0.001	2.4	0	0.00048

be carried out in a similar manner to the first, mixing 5 c.c. of the methyl alcohol solution with 5 c.c. of persulphate in concentrations varying only between the two above-mentioned limits. The number of mixtures in this series can be varied according to the degree of accuracy which is desired in the estimations. If, for example, it was found that the mixture of 5 c.c. methyl alcohol solution mixed with 5 c.c. of 4 per cent. persulphate gave a negative reaction for formaldehyde in the first series of experiments, whilst the solution mixed with 5 c.c. of a 3 per cent. solution gave a positive reaction, in the second series of experiments the methyl alcohol solution was mixed with ammonium persulphate solutions varying only between 3 and 4 per cent. For each concentration of methyl alcohol, therefore, there corresponds a definite concentration of the persulphate which is just sufficient, under the specified conditions of experiment, to oxidise all the formaldehyde which is formed in the reaction.

In the table on p. 165 are given the concentrations of methyl alcohol which are completely oxidised (so as to give a negative formaldehyde reaction) by concentrations of persulphate varying from 0.001 to 5 per cent.

The above table represents the final results of a detailed series of experiments, of which one is given *in extenso* to illustrate more clearly the method employed.

To determine the concentration of the solution of methyl alcohol which is just oxidised by a 5 per cent. persulphate solution, so that no formaldehyde is present after heating the mixture for ten minutes:

First Series :

				Reaction (Formaldehyde).
5 c.c. of this 5 per cent. persulphate solution +				
5 c.c. of 1 per cent. solution of methyl alcohol	...	...	strong pink	
4 c.c. " " "	1 c.c. H ₂ O	...	pink	
3 c.c. " " "	2 c.c. H ₂ O	...	"	
2 c.c. " " "	3 c.c. H ₂ O	...	"	
1 c.c. " " "	4 c.c. H ₂ O	...	0	

Hence the concentration of methyl alcohol solution, 5 c.c. of which is completely oxidised by 5 per cent. persulphate, lies between $\frac{1}{5}$ and $\frac{2}{5}$ of 1 per cent.

To determine the concentration more accurately, the following second series of experiments was carried out:

				Reaction (Formaldehyde).
5 c.c. persulphate solution +				
1.5 c.c. of 1 per cent. solution of methyl alcohol,	3.5 c.c. H ₂ O	...	faint pink	
1.45 c.c. " " "	3.55 c.c. H ₂ O	...	just pink	
1.4 c.c. " " "	3.6 c.c. H ₂ O	...	0	
1.3 c.c. " " "	3.7 c.c. H ₂ O	...	0	
1.2 c.c. " " "	3.8 c.c. H ₂ O	...	0	
1.1 c.c. " " "	3.9 c.c. H ₂ O	...	0	

Thus if the end point is taken to be that concentration which just gives the pink formaldehyde reaction after heating for ten minutes with the persulphate solution, it will be found that 5 per cent. of the latter just oxidises 5 c.c. of a solution containing

$\frac{1.45}{5} \times 1$ per cent. = 0.29 per cent. methyl alcohol. The percentages of methyl

alcohol corresponding to the various concentrations of persulphate are given in the final column of Table I. It is obvious that the concentration of a persulphate solution just necessary to oxidise a given concentration of methyl alcohol is a linear function of that concentration, and if c be the concentration (per cent.) of the persulphate, x that of the methyl alcohol, and k a constant, then $x = kc$. If this be calculated from the above table (neglecting the data for percentages of persulphate below 0.25 per cent.) k will be found to be $= 0.059$. These data were obtained with a methyl alcohol sample which had the correct boiling point, after careful fractionation over lime, and with a sample of persulphate in which the amount of ammonium persulphate was estimated by warming with a definite amount of standardised ferrous sulphate solution and determining the amount of the latter oxidised by titration against permanganate solution; this sample was found to contain 98.6 per cent. ammonium persulphate. In a series of experiments carried out with another sample of persulphate which was less pure, the constant k was found to be 0.054. In carrying out the estimation of methyl alcohol by the method above described, with any given sample of persulphate, it is best to standardise 5, 4, or 3 per cent. solutions against methyl alcohol and to determine directly the constant k . Only a few experiments will be necessary, as the results will only differ slightly (by fractions of a c.c.) from the numbers given in Table I., column 3.

B. Estimation of Methyl Alcohol in Ethyl Alcohol.

An attempt was made to estimate methyl alcohol in ethyl alcohol by the same method as that employed for estimating the methyl alcohol in water. This was found, however, to be impracticable, and there is no preferential oxidation of the lower alcohol by persulphate. It was found, for example, that the amounts of the latter necessary to oxidise to the end point (disappearance of formaldehyde) did not differ, outside the limits of experimental error, with ethyl alcohol mixtures containing 4 and 5 per cent. of methyl alcohol. Another method had therefore to be employed. This consisted in treating a persulphate solution with an excess of the mixture of alcohols, and examining the products produced in the initial stages of oxidation. It was then found that, under conditions such as these, the larger the amount of methyl alcohol present, the greater the amount of formaldehyde present, when the limited amount of persulphate used was entirely decomposed. The amount of formaldehyde produced under the conditions of the experiment could then be estimated colorimetrically. The estimations were carried out as follows: Ten c.c. of the ethyl alcohol containing methyl alcohol are diluted with 50 c.c. of water. Five c.c. of this are then mixed with 5 c.c. of a 1 per cent. solution of ammonium persulphate in a test-tube; this is provided with a short air condenser and heated in a boiling water-bath for ten minutes. At the end of this period, 1 c.c. of the mixture is added to 1 c.c. of a 1 per cent. solution of phenyl hydrazine hydrochloride, *with which it is heated in a boiling water-bath for five minutes*. After cooling, 1 c.c. of a 2.5 per cent. solution of potassium ferricyanide is added, and then 3 c.c. of concentrated hydrochloric acid. A pink colour is thereby produced when methyl alcohol is present, which can be compared with the colours produced in a similar way from samples of

ethyl alcohol containing known amounts of methyl alcohol. Experiments have been carried out with mixtures of ethyl alcohol and methyl alcohol, when the latter varied from 0.5 to 5 per cent., and it is possible by this rough comparison to estimate the percentage of methyl alcohol when contained within these limits to within 1 per cent. A more accurate estimation can be obtained when a colorimeter is employed. The method of carrying out the more accurate determination is described below.

It will be noticed, in the directions given above, that the reaction mixture is heated with the solution of phenylhydrazine hydrochloride before the other reagents are added, when carrying out the test for formaldehyde. The reason of this is that during the reaction certain quantities of the ethylal $\text{HCH}(\text{OC}_2\text{H}_5)_2$, and probably of the methylal $\text{HCH}(\text{OCH}_3)_2$, are formed during the heating, and the full pink colour is not given by formaldehyde combined in these ways, unless the mixture is heated with the solution of phenyl hydrazine hydrochloride before adding the other reagents. (Compare Schryver *loc. cit.*)

In carrying out the more accurate colorimetric estimations, the pink test solutions prepared by the method described above were diluted with 70 c.c. of water, and the colour was then compared with a standard prepared in an exactly similar way from a sample of ethyl alcohol containing a known amount of methyl alcohol. When a colorimeter of Schmidt and Haensch was used, differences could be detected between samples of ethyl-methyl alcohol mixtures in which the amounts of methyl alcohol differed by only 0.2 per cent. Possibly, with a more modern form of colorimeter, more accurate estimations would be possible. The researches on the determination of the amounts of methyl alcohol in ethyl alcohol have, up to the present, been confined to mixtures containing between 0.5 and 5 per cent. of the former.

The sample of ethyl alcohol gave a faint formaldehyde reaction when oxidised, and the oxidation products were tested for methyl alcohol in the manner described above. Whether this is due to the fact that it contains small amounts of the lower alcohol, or to the fact that formaldehyde is formed in small amounts by the oxidation of ethyl alcohol, it is impossible at the present to say.

C. Estimation of Methyl Alcohol in Acetone.

It was found that a similar method could be applied to the estimation of methyl alcohol in acetone to that employed for estimating this alcohol in water, as the acetone is only oxidised to a slight extent by persulphate. In the former case, however, there were certain difficulties in carrying out the estimation, which have necessitated, when the percentage of methyl alcohol present is small, certain minor modifications in the process. It was found when the acetone contained 4 per cent. or more of methyl alcohol that, as a certain concentration of persulphate was exceeded, there was a sudden transition from the bright pink colour yielded by the formaldehyde test to a light pink, and this latter colour persisted even when the above-mentioned concentration of persulphate was considerably above this transition point. This phenomenon is probably due to the formation of a condensation product of formaldehyde with acetone, which is very resistant to oxidation by persulphate. When the amount of methyl alcohol in acetone falls below 4 per cent., the transition

point is not very marked. At 4 per cent. and over, when higher concentrations of persulphate must be used, the point is unmistakable; for each given concentration of methyl alcohol in acetone, under the conditions of experiment described below, there corresponds a given concentration of persulphate, a small addition to which causes a sudden transition from a bright pink to a light pink, when the formaldehyde test is applied. By this method it is easy to determine to within 0.2 per cent. the percentage of methyl alcohol in acetone when the former varies between 4 and 20 per cent. (above the latter limit no experiments were carried out). When the percentage is below the former limit it can be determined approximately by comparing the colour reactions with those produced by acetone containing known amounts of methyl alcohol and when the experiments are carried out under the same conditions.

As an alternative method, when results accurate to within 0.2 per cent. can be obtained, the acetone mixture containing the lower percentage of methyl alcohol can be mixed with one containing a known higher percentage, so as to bring the total in the mixture to more than 4 per cent., and then carrying out the estimation of the methyl alcohol in this mixture by the method described in detail below, subsequently deducting the amount of added methyl alcohol from the result. A convenient method of carrying out the process is to add to the acetone-methyl alcohol mixture (containing the unknown amount of methyl alcohol) an equal volume of acetone containing 10 per cent. (by volume, as the specific gravities of methyl alcohol and acetone differ only very slightly) of methyl alcohol. By subtracting 5 from the percentage found in this mixture, and doubling the number thus obtained, the percentage of methyl alcohol in the mixture under investigation can be calculated.

The actual determination was carried out in the following manner: 5 c.c. of the acetone containing methyl alcohol under investigation were diluted to 250 c.c. with

TABLE II.

Per Cent. MeOH in Methyl-Alcohol Acetone Mixture.	Formaldehyde Reaction after Heating with Persulphate Solutions of the following Concentrations—				
	5 Per Cent.	4 Per Cent.	3 Per Cent.	2 Per Cent.	1 Per Cent.
10	0	?	+++	+++++	+++++
9	0	?	+++	+++++	+++++
8	0	0	+	+++	+++++
7	0	0	?	+++	+++++
6	0	0	0	++	+++++
5	0	0	0	+	++++
4	0	0	0	?	+++
3	0	0	0	0	++
2	0	0	0	0	+
1	0	0	0	0	?

water. Quantities each of 5 c.c. of this mixture were introduced into a series of test-tubes ($6 \times \frac{3}{4}$ ") containing 5 c.c. of ammonium persulphate solutions of varying concentrations. The test-tubes were provided with corks containing short air-condensers, and were then introduced into a water-bath containing boiling water in which they were kept immersed for ten minutes. At the end of this period each mixture was shaken, and the formaldehyde test was applied (using 1 c.c. of the hot solution) in the manner already given under the description of the determination of methyl alcohol in water. By using persulphate solutions of 1, 2, 3, 4, 5 per cent., an approximate estimation of the methyl alcohol can be made, as will be obvious from the table given on p. 169.

In the following table are given the percentages of persulphate in 5 c.c., which, when mixed with 5 c.c. of the diluted methyl alcohol acetone mixtures, just gave the positive reaction described above, which corresponds to the end point.

TABLE III.

Per Cent. CH_3OH in Original Mixture.	Per Cent. Concentration of Persulphate in 5 c.c. added.	Per Cent. CH_3OH in Original Mixture.	Per Cent. Concentration of Persulphate in 5 c.c. added.
20.0	6.4	8	2.56
17.5	5.8	7	2.24
15.0	4.8	6	2.04
12.5	4.0	5	1.7
10.0	3.2	[4	1.45]
9.0	2.9	—	—

The above researches were carried out for the Food Investigation Board.

Conclusions.—(1) A method for estimation of methyl alcohol in water is described, the essential principle of which consists in determining the concentration of ammonium persulphate necessary to destroy completely the formaldehyde formed in the initial stages of the oxidation process under certain specified conditions of experiment. For the detection of formaldehyde, the method previously described by one of the authors has been employed.

(2) By a slight modification of this method the amount of methyl alcohol in acetone can be estimated.

(3) To determine the amount of methyl alcohol in ethyl alcohol, the mixture of alcohols is partially oxidised by a relatively small amount of persulphate (the alcohols being in excess), and the formaldehyde formed under these conditions is estimated colorimetrically.

NOTE ON OPIUM POISONING.

By E. H. HANKIN, M.A., D.Sc., AND D. CHATTERJI.

DURING the last three years stomachs, with their contents, of fifty-three persons have been examined in this laboratory, in which the history of symptoms and other evidence obtained by the police indicated that opium was the cause of death.

The results of analysis were as follows: In one case no opium was detected, but opium was found in the viscera of another person who had been poisoned at the same time; in four cases the results of analysis were consistent with the presence of traces of opium; in twenty cases traces only of opium were detected; in nine cases opium was detected in small quantity; in ten cases opium was detected in medium quantity; in nine cases opium was detected in quantity.

By the term "small quantity" we mean that the residue obtained from a Stas-Otto extract gave colour reactions about as strongly as would the Stas-Otto extract of 7 mgrms. of Indian opium of 2 per cent. morphine content. The term "medium quantity" similarly corresponds to about 15 mgrms., and "detected in quantity" corresponds to 20 or more mgrms.

Thus there was a clearly negative result in one case. Out of the remaining fifty-two cases, in twenty-four opium was either detected in traces or the tests were responded to so faintly that no definite statement could be made as to the absence or presence of the poison. That in such a large proportion of the cases opium should only be detected in traces appears to be in accord with results obtained by other observers (see Witthaus, *Manual of Toxicology*, second edition, p. 980).

The only tests we have found to be of use in testing for opium in viscera are the following:

(1) *The Porphyroxin Test*.—We have carried out this test on several thousand extracts of viscera and other substances without ever getting a well-marked reaction, except in cases in which it was probable, on other grounds, that opium was present. On the other hand, a faint pinkish colour occurs not infrequently in the absence of opium.

(2) *The Husemann Reaction*.

(3) *The Urotropin or Formaldehyde Reaction*.

The last-mentioned test has been used as a colorimetric test for morphine in viscera (ANALYST, 1917, 42, 227). But, in our experience, there are grounds for doubting whether either this test or any other known to us can be depended on to give a reliable colorimetric result. The viscera are always sent to us preserved in alcohol. In the hot Indian climate it often happens that much decomposition has set in before the viscera are placed in alcohol. With such viscera we find that if an extract responds strongly to one of the above three tests there is no certainty that it will respond strongly to the others. The extent of this discrepancy is shown by the following table. It refers to analyses of thirty-one stomachs, three specimens of vomit, one of liver, and one of urine, a total of thirty-six specimens. In each case the history of symptoms, etc., had pointed to opium poisoning:

Reaction given by the—		Reactions given in the same cases by—	
Urotropin Test.		Porphyroxin Test.	Husemann Test.
Strong in eight cases ...	Strong	2	7
	Medium	5	0
	Negative or doubtful	1	1
Medium in sixteen cases	Strong	4	3
	Medium	12	13
	Negative or doubtful	0	0
Doubtful in six cases ...	Strong	0	1
	Medium	5	4
	Negative or doubtful	1	1
Negative in six cases ...	Strong	0	0
	Medium	6	6
	Negative or doubtful	0	0

Thus, in eight cases in which the urotropin reaction was strong, the porphyroxin reaction was only given strongly in two. In six cases the urotropin test gave a negative result, and in each of these the extract reacted with medium strength to the porphyroxin and Husemann tests. There can be little doubt that the cause of the occasional failure of the urotropin test is the inevitable presence of impurities in the extracts. It appears to be particularly difficult to get a pure extract in cases of opium poisoning. If an extract has a yellowish colour there is a presumption, in our experience, that it is going to respond strongly to the tests for opium.

When dealing with highly decomposed viscera preserved for a fortnight or more in alcohol, no amount of washing with ether or with ether and chloroform will so purify the acid solution that it will not allow impurities to pass into the solvent when made alkaline. Repeated washing of the acid solution with ether is likely to result in the porphyroxin test yielding a negative result, and a valuable piece of evidence will thus be lost. Hence, in carrying out the Stas-Otto process, we wash the acid solution once only with ether. The solution is then made alkaline and extracted with ether and chloroform. It is advisable to add these solvents separately. One part of chloroform is first added, then about three parts of ether, then a piece of litmus paper and, lastly, a few drops of ammonia. The mixture is then immediately shaken. We find that chloroform and ether thus used yield a purer extract than either an ethyl acetate-ether mixture or amyl alcohol.

NOTE.

The Editor desires to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

DETECTION OF COAL-TAR DYES IN BUTTER AND MARGARINE.

THE presence of these dyes can be readily detected by the fact that they are more stable to heat than vegetable butter-dyes or the natural colouring matter in butter.

The author has found on heating the clear filtered fat from butter or margarine to a temperature of 180° to 190° C. that natural butter colour and vegetable colours are destroyed, whilst coal-tar colours are not.

The test is carried out as follows: About 1 c.c. of the clear filtered fat (this is separated at a temperature not above 100° C.) is placed in a test-tube and immersed in an oil-bath which is raised to about 185° C. The test-tube is removed occasionally, shaken and replaced. In the absence of coal-tar dye the fat will become colourless within ten minutes; if the fat remains coloured, then coal-tar dye is present.

It has been found that butter-fat separated at temperatures above 100° C. will not become colourless on heating.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Modification of the American Official Chlorine Method for Feeds, Fæces, and Urine. J. O. Halverson and E. B. Wells. (*J. Biol. Chem.*, 1920, **41**, 205-208.) —Reasons are given for the inconsistent results obtained by the use of the official Volhard method, and the following modification is described. The alkaline ash is digested with dilute nitric acid, filtered and washed with hot water, the total volume of filtrate not exceeding 75 c.c. This solution is then boiled, $\frac{N}{50}$ silver nitrate gradually added in excess, and the boiling continued until the volume is reduced to 25 or 30 c.c. When cold, the solution is transferred to a 100 c.c. flask, made up to the mark, mixed, and, after standing for three hours, is filtered. Ninety-five c.c. are measured by a pipette, 2 c.c. of ferric nitrate indicator (prepared by boiling 125 c.c. concentrated nitric acid with 325 c.c. water until colourless, and adding 50 c.c. of saturated ferric nitrate solution) added and titrated with $\frac{N}{10}$ ammonium thio-cyanate, using a burette capable of being read to 0.01 c.c. A table is given showing results obtained on corresponding samples by the above, and by the official method, indicating that the former yields slightly higher (0.4 to 3 per cent.) values. Tests

made on salts of known chlorine content show that the proposed method yields a maximum error of ± 0.7 per cent., whilst the official method gives deviations of from 1 to 2 per cent.

T. J. W.

Identification of Citric Acid in the Tomato. R. E. Kremers and J. A. Hall. (*J. Biol. Chem.*, 1920, 41, 15-17.)—Strained tomato-pulp was saturated with calcium hydroxide and the calcium citrate obtained precipitated by boiling. The precipitate is not the pure calcium salt, since this requires 21.4 per cent. of calcium and only 15.6 per cent. was found. This impure calcium salt does not yield crystals when brought into reaction with phenacyl bromide; its purification is effected by conversion into the sodium salt with boiling sodium carbonate, and the calcium citrate is then re-precipitated by adding ammonium chloride and calcium chloride in excess to the solution of the sodium salt, followed by boiling and filtering whilst hot. The calcium salt is dissolved in 50 per cent. acetic acid, from which solution the lead salt is precipitated by basic lead acetate, the lead salt being then decomposed with sulphuretted hydrogen. The citric acid solution is filtered from the lead sulphide, concentrated in vacuo and crystallised in a vacuum desiccator. The triphenacyl ester was then prepared as follows: 0.7382 grm. of citric acid dissolved in 9 c.c. of water was neutralised with 0.5389 grm. of sodium carbonate; 1.61 grm. of phenacyl bromide dissolved in 18 c.c. of alcohol were added, and the mixture was boiled under a reflux for three hours. The reaction mixture was crystallised, the yield was 0.52 grm., melting at 104 to 105° C. Mixed with ester from U.S.P. sodium citrate, the m.-pt. was 104 to 105° C. Therefore, the presence of citric acid in the tomato juice has been shown by means of its triphenacyl ester.

H. F. E. H.

Estimation of Crude Fibre. O. Nolte. (*Zeitsch. anal. Chem.*, 1919, 58, 392-397.)—In the case of soft substances, the fineness of the powder has but little effect on the quantity of crude fibre obtained by the usual method of analysis; but with hard substances, such as buckwheat shells, barley chaff, etc., the coarse powder (larger than 2 mm.) yields about 4 to 5 per cent. more crude fibre than does the finely powdered material. Removal of fat before the acid and alkali treatment is unnecessary even in the case of fatty materials such as rape seed.

W. P. S.

Yield of Hydrocyanic Acid by *Phaseolus lunatus* Beans. H. Lührig. (*Chem. Zeit.*, 1920, 44, 166-167.)—The quantity of hydrocyanic acid found in nine samples of *Phaseolus lunatus* beans varied from 5.1 to 23.4 mgrms. per 100 grms. of beans. Treatment of the bean mash with emulsin did not materially increase the yield of hydrocyanic acid. The beans, after they had been steeped in water and then boiled for three hours, were free from bitter taste and did not produce any injurious effect when eaten in considerable quantity. (Compare also ANALYST, 1917, 42, 174.)

W. P. S.

Detection of Invertase in Honey. A. Caillas. (*Comptes rend.*, 1920, 170, 589-592.)—By precipitation with alcohol the author has separated from honey a substance which, by its action on cane sugar, was proved to be invertase; the yield was

0.049 per cent. of the honey. The presence of invertase in honey would explain the gradual disappearance of the cane sugar in honey when the latter is kept for some considerable time.
W. P. S.

Sugar in Oat Straw and Cattle Foods. S. H. Collins and A. Spiller. (*J. Soc. Chem. Ind.*, 1920, 39, 667.)—Many of the commoner cattle foods naturally contain sugar which is valuable in making them more palatable. The superiority of early-cut over late-cut oat straw probably depends on the higher sugar content of the former; by fermentation, however, much of the sugar may be lost by conversion into alcohol, lactic acid and butyric acid. Earth nut cake contains as much as 8.46 per cent. of sugar, all non-reducing. The total sugar in oat straw cut early ranges from 5.00 to 6.47 per cent., most of it being in the form of invert sugar. This sugar decreases considerably on storage, and in some cases disappears altogether. For the estimation, 5 grms. of the fodder are extracted with ether, and treated with 5 grms. of lead acetate and 250 c.c. of water. The mixture is allowed to stand overnight, then filtered, and 50 c.c. of the filtrate are acidified with 5 c.c. of strong hydrochloric acid, and warmed slowly so that it reaches 60° C. after twenty minutes. Any lead chloride is filtered off, dry sodium carbonate is added in excess, and the filtered liquid titrated with Fehling's solution. Some difficulty is experienced in obtaining clear solutions, and the cuprous oxide tends to remain persistently in suspension.

J. F. B.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Detection and Characterisation of Dextrose in Plants by a New Biochemical Method. E. Bourquelot and M. Bridel. (*Compt. rend.*, 1920, 170, 631-635.)—The ordinary methods of identification, namely reduction of Fehling's solution, fermentation by yeast and the formation of glucosazone, are not sufficient for the definite characterisation of dextrose, since other monose sugars possess the same properties. The authors describe a method for the identification of dextrose which is perfectly specific. This test consists of a reversal of the method for the detection of glucosides by emulsin. The hydrolytic action of emulsin is reversible, and if this enzyme be added to a solution of dextrose in an alcohol a glucoside is produced and the optical activity of the liquid becomes levorotatory in proportion to the quantity of glucoside formed. The reaction has been demonstrated in the following manner: A solution is made in 70 per cent. methyl alcohol of 2.0565 grms. of dextrose per 100 c.c. The rotation of this solution in the 200 mm. tube is +2° 20'. To this is added 0.5 gm. of emulsin, and the mixture is allowed to stand at the temperature of the laboratory. At the equilibrium point the rotation will be found to have become -50' corresponding to a displacement to the left of 3° 10', from which it is calculated that 1.6978 grms. or 82.55 per cent. of the dextrose has combined with the alcohol. Moreover, the methylglucoside is easily isolated by evaporating the solution to dryness under reduced pressure and extracting the residue with boiling ethylacetate, from which the glucoside crystallises on cooling. Further experiments with mixtures of dextrose with mannose, fructose, and arabinose showed that only the

dextrose was etherified, and, under the conditions above mentioned, 83 per cent. of the dextrose was always combined. The specific rotatory power of β -methylglucoside is -32.5° . The method has been applied for the identification of dextrose in juniper berries and in the glucoside loroglossin obtained from *Loroglossum hircinum* Rich.

J. F. B.

Comparative Study of Hæmoglobin Determination by Various Methods.

F. S. Robschelt. (*J. Biol. Chem.*, 1920, **41**, 209-226.)—The history of various methods of hæmoglobin determination is given, their disadvantages indicated, and tables showing comparative results obtained by the different methods are provided. A modification is described, which consists of the application of Palmer's procedure for comparison of the colour of carbon-monoxide hæmoglobin solutions (*J. Biol. Chem.*, 1918, **33**, 119) to Sahli's acid hæmatin method (*Lehrbuch der klin. Untersuchungs-Methoden*, Leipsic, Fourth Edition, 1905). The oxygen capacity of dog's blood is determined by Van Slyke's method (*J. Biol. Chem.*, 1918, **33**, 127), and the hæmoglobin content calculated. A 20 per cent. suspension is prepared, such that a 1 per cent. dilution would read 100 per cent. hæmoglobin. The undiluted standard retains its original colour at least eight months if kept in an ice-chest. One c.c. of this solution is pipetted into about 50 c.c. of $\frac{N}{10}$ hydrochloric acid, mixed, the volume made up to 100 c.c. with $\frac{N}{10}$ hydrochloric acid, and left to stand in an ice-chest for one hour. The flask is then thoroughly shaken and the solution transferred to a colorimeter tube. The blood under examination is treated in the same manner, and the colour compared with the standard. The illuminant may be daylight or a nitrogen-filled electric bulb, fitted with a "Daylite" colour screen. The dilute acid hæmatin solutions slowly fade after about a week, even if kept in an ice-chest. Similar results are obtained whether sheep's, goat's, or human blood be employed as the standard.

T. J. W.

Determination of Methæmoglobin in Blood. **W. C. Stadie.** (*J. Biol.*

Chem., 1920, **41**, 237-241.)—Two c.c. of oxalated whole blood are placed in a 100 c.c. flask, 50 c.c. of water added, and 0.5 c.c. of a 3 per cent. potassium ferricyanide solution is then run in, and the flask allowed to stand about twenty minutes, after which 5 c.c. of a 0.1 per cent. potassium cyanide solution are added, and the mixture made up to 100 c.c. by the addition of water. The colour is then compared with a standard of known strength in a colorimeter, and the result represents the hæmoglobin plus methæmoglobin, which is expressed as grms. of "total hæmoglobin" per 100 c.c. of blood. *Preparation of Standard*: The hæmoglobin content of fresh whole oxalated or defibrinated blood free from methæmoglobin is determined gasometrically, 10 c.c. of the blood are hæmolyzed by the addition of about 300 c.c. of water, 2.5 c.c. of the above potassium ferricyanide solution are run in, and, after twenty minutes, 25 c.c. of the potassium cyanide solution are added, and the whole made up to 500 c.c. with water. The actual hæmoglobin present in the sample under examination is determined by aerating 4 to 5 c.c. of the blood in a funnel and determining the total oxygen content by the Van Slyke method. Dividing the figure representing this value by 1.84 gives the grms. of hæmoglobin per 100 c.c. of sample. The

methæmoglobin present is deduced by deducting the actual hæmoglobin content from the "total hæmoglobin." Tables are provided for the correction of gas volumes obtained in the gasometric determination of hæmoglobin, and results of methæmoglobin determinations made by the above method compared with the corresponding calculated values. These show satisfactory agreement. Attempts were made by the author to estimate methæmoglobin by conversion into carbonmonoxide-hæmoglobin, and also into acid hæmatin, but were unsuccessful.

T. J. W.

Estimation of Indole in Biological Media. H. F. Zoller. (*J. Biol. Chem.*, 1920, **41**, 25-36.)—Previous methods devised for the estimation of indole are discussed, and various difficulties met with in their application are pointed out. The present method is based on the results obtained in former work performed by the author upon the distillation of indole (Zoller: *J. Biol. Chem.*, 1920, **41**, 37-44) in combination with a modification of Nonnetta and Demanche's application of Baeyer's nitroso-indole reaction. To 100 c.c. of the medium under examination $\frac{N}{1}$ sodium hydroxide solution is added until the hydrogen ion concentration equals Ph. 9.2; this will be attained when a spot test shows a clear red colour with phenolphthalein, and no blue colour with thymolphthalein, both indicators having a concentration of 0.02 per cent. The solution is then distilled until about 10 c.c. remain, and the distillate is diluted to 100 c.c. A standard solution is prepared by dissolving 10 mgrms. of pure indole in 100 c.c. of water, and varying portions containing 0.02 to 0.20 mgrm. of indole are run into a series of test-tubes uniform in diameter and colour, and each diluted to 10 c.c. To each tube 2 drops of 1 per cent. sodium nitrite solution and 5 drops of concentrated sulphuric acid are added; the contents are then gently mixed and allowed to stand about five minutes. The solution is extracted with three 3 c.c. portions of either isoamyl or isobutyl alcohol, and the coloured alcoholic extracts pipetted into another test-tube, and diluted to exactly 10 c.c. These standards are permanent for at least one week. An aliquot portion of the distillate, containing from 0.01 to 0.20 mgrm. of indole, is transferred to a tube and treated in the same manner as the standard solutions, the indole present being estimated by comparison of the red colour produced with those of the standards. A table is provided, showing that the results obtained by the author's method agree satisfactorily with those yielded by the β -naphtha-quinone-sodium-mono-sulphonate method of Bergeim (*J. Biol. Chem.*, 1917, **32**, 17), while the time occupied by the former determination is less than one-third that required for the latter. The new method is simple, reliable, and rapid, and requires no special apparatus.

T. J. W.

Estimation of Potassium and Sodium in Small Quantities of Blood. B. Kramer. (*J. Biol. Chem.*, 1920, **41**, 263-274.)—*Potassium*.—One c.c. of blood or 3 to 5 c.c. of clear plasma or serum is dried in a platinum dish on a steam bath, and finally at 110° C. for thirty minutes. The residue is gradually heated until completely incinerated, keeping the temperature as low as possible. On cooling, the ash is dissolved in 0.5 c.c. of water with one or two drops of glacial acetic acid and 0.5 to 1.0 c.c. of sodium cobalti-nitrite reagent (see below) are gradually added with stirring and the mixture left for ten minutes. The precipitate is filtered off through

a Gooch crucible provided with a thick asbestos felt, and washed with cold water until the washings are clear. The asbestos and precipitate are transferred to a beaker, 25 c.c. of $\frac{N}{100}$ potassium permanganate and 5 c.c. of 25 per cent. of sulphuric acid added, and the mixture heated on a water-bath for three minutes. Sufficient $\frac{N}{100}$ oxalic acid is run in to decolorise the solution completely, and this is immediately titrated with $\frac{N}{100}$ potassium permanganate to a permanent pink colour. The total volume of the potassium permanganate solution used minus the volume of oxalic acid solution added $\times 0.071 =$ mgrm. of potassium in the sample. Sodium cobalti-nitrite reagent: (A) 50 grms. of cobalt nitrate are dissolved in 100 c.c. of water, and 25 c.c. of glacial acetic acid added (B) 50 grms. of pure sodium nitrite in 100 c.c. of water. Six volumes of A are mixed with ten volumes of B, and air bubbled through the mixture. The mixed reagent will keep for at least a month in an ice-box, and should be filtered before use.

Sodium.—The ash from 1 or 2 c.c. of material obtained as in the potassium estimation is dissolved in 0.5 or 1 c.c. of water and one drop of $\frac{N}{10}$ hydrochloric acid. Fresh 10 per cent. potassium hydroxide solution is added until slightly alkaline, and 15 c.c. of potassium pyroantimoniate reagent (see below) run in followed by one-fifth the total volume of absolute alcohol. The mixture is left to stand at least two hours, transferred to a moist asbestos Gooch filter, washed with 3 c.c. portions of 30 per cent. alcohol, dried at 110° C. and weighed. A blank estimation should always be made. Potassium pyroantimoniate reagent: 2 grms. of the salt are boiled with 100 c.c. of water until no more dissolves, 3 c.c. of 10 per cent. potassium hydroxide are added and the mixture stirred and filtered. This reagent should be freshly made.

The methods are usually accurate to within 3 per cent. The potassium content of normal human serum varies between 16 and 22 mgrms. per 100 c.c. and the sodium content, both in children and adults, between 280 and 310 mgrms. per 100 c.c.

T. J. W.

Estimation of Saccharin in Urine. G. S. Jamieson. (*J. Biol. Chem.*, 1920, 41, 3-8).—Urine is treated with a solution of normal lead acetate and filtered. The filtrate acidified with hydrochloric acid is extracted with ether, the ether evaporated and the saccharin again extracted from the residue with ether. The ether is removed, the residue fused with sodium carbonate and the sulphur determined as barium sulphate ($\text{BaSO}_4 \times 0.7844 = \text{saccharin}$). Ether is the best solvent (ANALYST, 1914, 39, 354) for the extraction, and good results are obtainable for amounts of saccharin varying from 1 mgrm. and upwards per 100 c.c. of urine. Measured quantities of a standard saccharin solution were added to urines, and the mixtures analysed with very accurate results, the average error when working with from 0.001 to 0.01 gm. of saccharin in 100 c.c. of urine being about 0.0001 gm. A blank determination usually yields about 0.7 mgrm. of BaSO_4 , which should be deducted from that found; the blank should, if possible, be performed on the urine of the subject to be experimented with. Working on 100 c.c. of urine, it was found that about 10 c.c. of a 20 per cent. normal lead acetate solution was a suitable amount to use; excess must be avoided, since precipitation of lead chloride on acidification interferes with the ether extraction; while too little lead acetate leaves matters

present which form emulsions with ether. It is essential to heat urines which contain albumen, almost to boiling for a few minutes before the addition of lead; the subsequent filtration is best effected by suction through a mat of paper pulp about 0.5 cm. thick; this generally occupies about one hour. The filtrate is acidified with 15 c.c. of concentrated hydrochloric acid, followed by 55 c.c. of ether. Elaborate details for the extraction with ether are given in the paper; about 2 grms. of sodium carbonate are used for the fusion in a nickel crucible of the residue finally obtained.

H. F. E. H.

ORGANIC ANALYSIS.

Estimation of Acetaldehyde in Paraldehyde. F. V. Bruckhausen. (*Apoth. Zeit.*, 1919, **34**, 428; *Chem. Zeit. Uebersicht*, 1920, **44**, 77.)—Romijn's method of estimating formaldehyde iodimetrically cannot be adapted to the estimation of acetaldehyde, since the latter reacts with iodine in alkaline solution to form not only iodoform and acetic acid, but also other compounds, so that varying amounts of iodine are consumed. Richter's method, however (*ANALYST*, 1912, **37**, 265), gives trustworthy results, especially when the acetaldehyde and sulphite solutions are cooled to 5° C. before the titration. Paraldehyde containing acetaldehyde consumes permanganate, since it contains hydrogen peroxide. There appears to be a proportional relationship between the amounts of acetaldehyde and hydrogen peroxide.

C. A. M.

Estimation of Dextrose in the Presence of Lactose. E. Hildt. (*Ann. Chim. anal. appl.*, 1920, **2**, 78-80.)—Lactose may be quantitatively hydrolysed by means of sulpho-aromatic acids, as used by Twitchell in the technical hydrolysis of fats. A suitable reagent consists of a solution of 180 grms. of benzene sulphonate (free from iron) in a litre of water containing 49 grms. of pure sulphuric acid. For the hydrolysis of 0.5 to 1 per cent. solution of lactose 20 c.c. of this solution are added, and the liquid heated for six hours in an oven at 95° to 98° C. In applying the method to urine 100 c.c. of the sample are first treated with 10 c.c. of neutral lead acetate solution, and a preliminary test made of the approximate amount of sugar. A volume of the liquid containing not more than about 1 gm. of sugar is mixed with 20 c.c. of the hydrolyzing solution, and made up to 100 c.c., and 50 c.c. of the mixture are heated for six hours at 95° to 98° C. The remainder of the liquid is used for the direct titration of sugars by means of Fehling's solution diluted with 4 volumes of water and treated with 5 or 6 drops of sodium hydroxide solution. The hydrolysed liquid is cooled, made up to 50 c.c. and titrated in the same way, and the results expressed in terms of dextrose or anhydrous lactose, 0.051 gm. of which is equivalent to 0.071 gm. of hydrated lactose, or 0.048 gm. of anhydrous dextrose. From these data the amount of hydrated lactose, x , and anhydrous dextrose, y , in 100 c.c. of the urine may be calculated by means of the equations—

$$x = \frac{3.55}{n'} - \frac{3.55}{n}; \quad y = \frac{1.458}{n'} + \frac{3.34}{n},$$

where n and n' represent the numbers of c.c. of the liquid required to decolorise

10 c.c. of Fehling's solution before and after inversion. In each case these quantities of solution contain an amount of sugars equivalent to 0.051 grm. of inverted lactose.

C. A. M.

Influence of Ester Transposition in the Determination of Saponification Values. A. M. Pardee and E. E. Reid. (*J. Ind. and Eng. Chem.*, 1920, **12**, 129-133.)—In the estimation of the saponification value of essential oils it is usual to heat the solution under a reflux condenser, consisting merely of a thin walled glass tube about 1 metre long. The results, however, are frequently low and variable, and in the saponification of acetylated oils, for the estimation of the alcohols present, the error is very much greater. This is due to transposition of the alkyl radical of the ester with the alcoholic medium of the saponifying agent, which takes place at a faster rate than the saponification. The result is that a highly volatile ester is produced in the preliminary stages and methyl acetate or ethyl acetate escapes through the tube of the reflux condenser. For instance, in the saponification of benzyl acetate, an ester boiling at 206° C., the first stage consists in the formation of ethyl acetate, b.p. 77° C., or of methyl acetate, b.p. 54° C., if methyl alcoholic potassium hydroxide is used, and considerable losses may occur. In the saponification of benzyl formate the corresponding formates are produced having still lower boiling points. On the other hand, the saponification of methyl benzoate in methyl or ethyl alcoholic solution will not give rise to highly volatile intermediate products. The authors have investigated this reaction and have shown how unreliable are the results obtained in certain cases when using methyl or ethyl alcoholic solutions. Normal butyl alcohol appears to be the most satisfactory medium in which to carry out the saponification of the esters in essential oils.

J. F. B.

Estimation of Halogen in Organic Substances. W. A. Van Winkle and G. McP. Smith. (*J. Amer. Chem. Soc.*, 1920, **42**, 333-347.)—The substance, volatilised with air, is drawn through a heated quartz tube, and the combustion products are absorbed in alkaline sodium sulphite solution; the excess of sulphite is then oxidised with permanganate, and the halogen estimated by Volhard's method. The quartz combustion-tube is about 24 inches long and is slightly inclined, the lower end being bent downwards and connected with the absorption apparatus; the other end of the tube is connected with the volatilisation tube. This consists of a deep test-tube with a side tube near the top and another lower down, the latter reaching well into the end of the combustion tube. The test-tube is closed with a rubber stopper carrying a glass rod for breaking the capsule containing the liquid under examination; a pad of glass wool protects the rubber stopper from attack by the substance. The combustion tube is heated by four wing-top Bunsen burners, an inverted V-shaped asbestos reflector being placed above the tube. The absorption apparatus consists of a vertical tube partially filled with clean sand; the lower end of the tube passes through a cork into a flask connected with a suction pump, whilst the upper end is closed with a rubber stopper through which pass the end of the combustion tube and a short tube connected with a tapped funnel containing the alkaline sodium sulphite solution. During the combustion, a current of air is drawn

through the apparatus and the sodium sulphite is admitted slowly, the top of the absorption tube being so arranged that the solution first flows on to the end of the combustion tube and drips from the latter on to the sand, passing through this into the flask. A secondary current of air is admitted directly into the combustion tube through a side tube. For the analysis of non-volatile substances the volatilisation part of the apparatus and the combustion tube are made in one piece, so that the substance may be heated and burnt in the volatilisation tube and the gases then passed into the combustion tube. The apparatus yields trustworthy results with such substances as ethyl iodide, butyl bromide, benzal chloride, chloroform, *m*-iodobenzoic acid, *p*-bromaniline, etc.

W. P. S.

Study of the Nephelometric Values of Cholesterol and the Higher Fatty Acids.—II. F. A. Csonka. (*J. Biol. Chem.*, 1920, **41**, 243-249.)—A continuation of work previously described by the author (*J. Biol. Chem.*, 1918, **34**, 577). Oleic acid and cholesterol were selected for the investigation because these show the largest difference in their nephelometric values. *Technique A*: Turbidity was produced by the addition of hydrochloric acid after water had been added to the alcoholic solution of the fat. *Technique B*: The alcoholic solution of the fat was added to water and then hydrochloric acid added as above. In Series I. oleic acid and cholesterol were used unmodified, but in Series II. the above substances were saponified by adding 2 c.c. of $\frac{N}{10}$ sodium hydroxide and placing in a boiling water-bath for fifteen minutes. The residue was warmed with 4 c.c. of 95 per cent. alcohol to dissolve the fatty material, and the final volume made up to 50 c.c. with an acidity of $\frac{N}{10}$ hydrochloric acid. For further details the previous paper should be consulted. The nephelometric value varies with the substance employed, the technique adopted, the presence or absence of a protective colloid, and, in some cases, with the period of time elapsing before taking the reading. Tables and curves are given showing the effects of these variables.

T. J. W.

Method of Preparing Phloroglucinol Reagent for the Kreis Test.
H. W. Dixon. (*J. Ind. and Eng. Chem.*, 1920, **12**, 174.)—The Kreis test has been found to be useful for the detection and estimation of rancidity in fats, and the phloroglucinol reagent may be prepared in the following manner: 100 grms. of sodium hydroxide are dissolved by heating slowly in a metal vessel with 40 c.c. of water. The melt is cooled to such a temperature that a few crystals of resorcinol dropped into the liquid do not become brown, 15 grms. of resorcinol are then stirred in. The basin is covered with a watch-glass to exclude air, and heated for two to three hours on a sand-bath, taking care not to char the product, until the melt is a dark chocolate-brown. If the reaction is not carried to completion, unchanged resorcinol will interfere with the colour of the Kreis test. The melt is cooled with exclusion of air, then dissolved in about 500 c.c. of water with gentle heat if necessary. The solution is transferred to a beaker, cooled in ice and acidified by the cautious addition of concentrated hydrochloric acid. The acid liquid is extracted repeatedly by shaking with 100 c.c. portions of ether in a separating funnel. The ether extracts are combined and decolorised by shaking with animal charcoal. The filtered ether

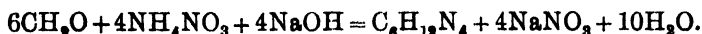
extract should be kept as a stock solution. To prepare the Kreis reagent a portion of the stock solution should be diluted with ether until it gives a good reaction with a strongly rancid fat, but a less intense reaction on further dilution. Fifteen grms. of resorcinol should yield 10 litres or more of Kreis reagent. J. F. B.

Change of Refractive Indices of Fixed Oils with Temperature. A. F. Joseph. (*J. Soc. Chem. Ind.*, 1920, 39, 66T.)—In a paper by C. H. Wright (ANALYST, 1920, 52) a formula was given for calculating the refractive index of an oil at the standard temperature of 40° C. from observations made at any other temperature. The author points out that the new formula for temperature correction was hardly necessary, since it shows no advantage in accuracy or simplicity as compared with Tolman and Munson's figures (*J. Amer. Chem. Soc.*, 1902, 24, 754), which showed that dn/dt for different oils is very nearly a constant of value 0.000365. The correction of refractive index for temperature should therefore be made by the simple method of adding or subtracting 0.00036 for every degree of temperature correction.

J. F. B.

INORGANIC ANALYSIS.

Formaldehyde Method for Determining Ammonium Nitrate. J. T. Grissom. (*J. Ind. and Eng. Chem.*, 1920, 12, 172-173.)—With the object of standardising a rapid method for the control of the manufacture of ammonium nitrate the author has investigated the reaction by which hexamethylene-tetramine is produced with formaldehyde, resulting in a corresponding neutralisation of sodium hydroxide. The equation representing the reaction is:



A 20 per cent. solution of formaldehyde neutralised to phenolphthalein is added to the ammonium nitrate solution, the mixture is warmed to 60° C., and titrated with $\frac{N}{3}$ sodium hydroxide, using phenolphthalein as indicator. This is better than employing an excess of standard sodium hydroxide and titrating back with acid, in which case the results are unreliable, owing to the formation of formic acid. The direct titration method, using $\frac{N}{3}$ sodium hydroxide, gives consistent results which are always about 0.25 per cent. low. This method is recommended for rapid control work, and may be made quite accurate by the addition of a constant correction factor to the results. The factor is determined by performing a Kjeldahl estimation on a representative sample and comparing the result with that obtained by the formaldehyde method.

J. F. B.

Qualitative Analysis in the Presence of Phosphoric Acid. H. Remy. (*Zeitsch. anal. Chem.*, 1919, 58, 385-392.)—The procedure depends on the use of phosphoric acid itself as a group reagent; from an ammoniacal solution, iron, manganese, chromium, alkali-earths, and magnesium are precipitated as phosphates or double phosphates, whilst zinc, nickel, and cobalt form soluble complexes. After heavy metals have been removed by hydrogen sulphide the solution is boiled

to expel excess of hydrogen sulphide, filtered if necessary, and oxidised with nitric acid; the solution is then nearly neutralised with solid ammonium carbonate and poured, with stirring, into an equal volume of warm concentrated ammonia. The mixture is now treated with an excess of ammonium phosphate solution. The phosphate precipitate is collected, dissolved in a small quantity of warm dilute hydrochloric acid, and potassium sulphate solution is added. Barium, strontium, and a portion of the calcium are precipitated as sulphates, and may be separated and identified in the usual way after fusion with sodium and potassium carbonates. The filtrate from the alkali-earth sulphates is boiled with the addition of sodium acetate and ammonium chloride; iron, chromium, and aluminium are precipitated as phosphates, and manganese as ammonium manganese phosphate. The precipitate is collected on a filter, and ammonium oxalate is added to the filtrate (this is still an acetic acid solution); calcium is precipitated as oxalate, whilst magnesium may be precipitated by adding ammonia to the filtrate from the calcium oxalate precipitate. The phosphate precipitate containing iron and other metals is dissolved in hydrochloric acid, the solution heated and nearly neutralised with solid sodium carbonate, and then poured into a mixture of 20 per cent. sodium hydroxide solution and hydrogen peroxide; ferric hydroxide and hydrated manganese dioxide are precipitated, whilst chromium and aluminium remain in solution as chromate and aluminate respectively. After filtration, the iron and manganese may be identified by the usual reactions; aluminium is precipitated on boiling the filtrate with the addition of ammonium chloride, and chromium, already indicated by the yellow colour of the solution, is identified by the perchromic acid reaction. The filtrate from the first phosphate precipitate, and containing the zinc, nickel, cobalt, and alkalis, is heated and treated with ammonium sulphide, and the precipitated zinc, nickel, and cobalt sulphides are collected; zinc sulphide dissolves in dilute hydrochloric acid, whilst nickel and cobalt sulphides remain insoluble and may be identified in the usual way. In the filtrate from the sulphide precipitate, potassium may be identified by the perchloric acid test, and sodium by means of potassium pyroantimoniate. In the case of a mixture free from heavy metals, but containing chromates and permanganates, the preliminary treatment with hydrogen sulphide may be omitted; the chromium and manganese are then found in the solution containing the zinc, nickel, and cobalt phosphates. On treatment with ammonium sulphide they are precipitated as hydroxides and are dissolved, together with the zinc sulphide, by dilute hydrochloric acid. Zinc remains dissolved when this solution is heated with an excess of sodium hydroxide.

W. P. S.

Estimation of Carbon Dioxide, Oxygen, and Combustible Gases by Krogh's Method of Micro-Analysis. H. O. Schmit-Jensen. (*Biochem. J.*, 1920, 14, 4-24.)—Krogh has described a method of micro-analysis for volumes between 1 and 20 c. mm. (*Scand. Arch. Physiol.*, 1908, 20, 278; *Abderhalden's Biochem. Arbeitsmeth.*, 1915, 8, 495).—The principle of the method is based on the fact that small air-bubbles placed in a fluid behave much as semi-fluid fat drops, and easily allow transport from one place to another without disunion. For analysis an air-bubble is sucked up into a capillary tube of uniform bore and measured. The tubes

used by Krogh had a diameter of about 0.25 mm. Gas absorptions are performed one after another in a funnel at the lower end of the capillary tube. Each absorption being finished, the air-bubble is sucked into the capillary tube again and measured anew. Krogh applied this method to the estimation of carbon dioxide, oxygen, and, according to the present author (who acknowledges help from Krogh throughout his research), to carbon monoxide, although there is no mention of this in the papers cited above.

The author found it necessary to devise means for the analysis of gas originating from intestinal gas cysts and containing hydrogen and methane. It was found that Krogh's apparatus served for the estimation of carbon monoxide and hydrogen, using acid cuprous chloride solution, and Paal and Hartmann's solution of colloidal palladium and sodium picrate (*ANALYST*, 1910, **35**, 139) respectively as absorbents. For the estimation of methane it was necessary to devise micro-combustion apparatus. For this purpose the diameter of the capillary was increased so as to admit of the introduction of platinum wires. The diameter finally adopted was about 1 mm. Platinum wires could be introduced into tubes of smaller bore, but the insertion of them into bubbles of 5 c. mm. in tubes of the diameter used by Krogh was made impossible by the surface tension, the bubble escaping when meeting the wire. The apparatus finally adopted is fully described and illustrated in the text, as is a micro-receiver for small gas samples. Carbon dioxide is absorbed in the usual manner, oxygen by alkaline pyrogallol, carbon monoxide by acid cuprous chloride solution, hydrogen is burned with excess of oxygen below a visible heat and methane at a bright red heat. The quantity of gas taken for an estimation is about 80 c. mm., the accidental error of a single estimation ± 0.1 c. mm., and the systematic error in the case of oxygen, hydrogen, methane, and probably carbon monoxide negligibly small. The systematic error in estimations of carbon dioxide, which diffuses with enormous rapidity in most fluids, is relatively large, over +0.5 per cent. in the author's hands. For any one systematic operator, however, and any small range of concentration of carbon dioxide, it should be substantially a constant. G. C. J.

Effect of Chlorides on the Nitrometer Determination of Nitrates. M. T. Sanders. (*J. Ind. and Eng. Chem.*, 1920, **12**, 169-170.)—Determinations were made to ascertain the maximum quantity of sodium chloride which may be present in crude sodium nitrate without introducing a serious error in the estimation of the nitrogen content by means of the nitrometer. Mixtures of known quantities of sodium nitrate and sodium chloride were analysed in the nitrometer with all due precautions, and a curve was plotted showing the percentage error with increase in the proportion of chloride. It was found that the error was reasonably small until the proportions of the mixture rose to 3 mols. of chloride and 1 mol. of nitrate. At this point the curve makes a sharp break upwards, and the presence of more than 3 mols. of chloride to 1 of nitrate makes the estimation quite impossible. It is probable that nitrosyl chloride is formed, which attacks the mercury and liberates nitric oxide which, recombining with free chlorine, repeats the cycle until all the free chlorine is used up. In such a case hydrochloric acid exists in the gases in the measuring tube, and the result is too high. Hence it is concluded that if the percentage of sodium chloride present in the

dry crude nitrate exceeds 15–17, it is impossible to obtain results with the nitrometer which are accurate to 0.1 per cent. J. F. B.

Preparation of a Hydrochloric Acid Solution of Cuprous Chloride for Use in Gas Analysis. F. C. Krauskopf and L. H. Purdy. (*J. Ind. and Eng. Chem.*, 1920, 12, 158–161.)—A solution of cuprous chloride in excess of hydrochloric acid is recommended by Winkler for the absorption of carbon monoxide in gas analysis. Such a solution may be conveniently and rapidly prepared by dissolving cupric chloride in concentrated hydrochloric acid and reducing the cupric salt by the addition of stannous chloride. By using a slight excess of stannous chloride the cuprous salt is protected against oxidation during transference from one container to another. The presence of stannic and stannous chlorides in relatively large amounts in a hydrochloric acid solution of cuprous chloride does not impair its efficiency for the absorption of carbon monoxide. When the cuprous chloride solution has become saturated with carbon monoxide it may be restored by heating at 60°–70° C. for some hours. The whole of the carbon monoxide is thereby expelled and the regenerated solution is quite as efficient for the absorption of further quantities of carbon monoxide as the original. If a small amount of the copper has become oxidised during the treatment the solution may be decolorised by the addition of a few drops of concentrated stannous chloride solution. J. F. B.

Estimation of Nitrogen in Nitrocellulose and Inorganic Nitrates by the Nitrometer. E. G. Beckett. (*J. Chem. Soc.*, 1920, 117, 220–235.)—It is shown that the nitrometer method of estimating nitrogen in nitrocellulose invariably yields low results. The interval of time which is allowed to elapse between the introduction of the nitrate and sulphuric acid into the nitrometer and the shaking has a great influence on the results in the case of nitrocellulose; with inorganic nitrates the effect is less. The most trustworthy results are obtained by using 15 c.c. of 92.5 to 94.0 per cent. sulphuric acid, and allowing fifteen minutes to elapse between the introduction of the nitrocellulose and the shaking. In this case the error amounts to about 0.7 per cent. of the nitrogen present. W. P. S.

Modification of the Thompson Method for the Estimation of Acetic Acid in White Lead. L. McMaster and A. E. Goldstein. (*J. Ind. and Eng. Chem.*, 1920, 12, 170–171.)—The Thompson method is the most reliable one for the estimation of acetic acid in white lead, both for those samples which have not been ground in oil and for those from which the oil has been extracted. The principal drawback in the latter case is due to the difficulty of complete extraction and the distillation of fatty acids which give a turbid distillate and interfere with the titration of the acetic acid. This difficulty is entirely overcome by performing the distillation under reduced pressure. The procedure is as follows: 18 grms. of extracted white lead are placed in an ordinary 500 c.c. flask with 40 c.c. of syrupy phosphoric acid (85 per cent.) and 50 c.c. of water. The flask is heated directly and the material distilled down to a small bulk. Steam is then passed into the flask and distillation continued until about 600 c.c. of distillate are obtained. After adding

about 0.5 c.c. of phosphoric acid the distillate is transferred to a heavy-walled flask fitted with a two-hole stopper. Through one hole passes a tube with a very small capillary bore, through which a minute stream of air may be drawn in during the distillation. Through the other hole is passed a connecting bulb tube attached to a condenser. Distillation is conducted under reduced pressure, corresponding to about 150 mm. of mercury, using a conical filtering flask as a receiver. Distillation is stopped when 10 c.c. of the distillate required only one drop of $\frac{N}{10}$ alkali to produce a colour change in phenolphthalein. At no time should the volume of liquid in the distillation flask be allowed to fall much below 100 c.c., as phosphoric acid may then be drawn over. In this method the final distillate is always obtained clear, and the results are accordingly slightly lower than by the Thompson method, which always yields cloudy distillates. It is possible even to proceed to the analysis without previously extracting the oil.

J. F. B.

Determination of Oxygen by the Copper-Ammonia-Ammonium Chloride Reagent. W. L. Badger. (*J. Ind. and Eng. Chem.*, 1920, 12, 161-164.)—Hempel has described a reagent for the absorption of oxygen in gas analysis, consisting of spirals of metallic copper immersed in a liquid consisting of equal parts of saturated ammonium carbonate solution and ammonia of sp. gr. 0.93. The author has investigated this reagent, but found that it is not the most effective solution of its type for the quantitative absorption of oxygen. The most suitable reagent is made by saturating with ammonium chloride a mixture of one part of concentrated ammonia and one part of water. Such a solution when employed in the absorption pipette in the presence of copper spirals will absorb fifty to sixty times its volume of oxygen. It must then be changed, not because it fails to absorb the oxygen quantitatively, but because such a heavy precipitate is formed that the reagent becomes unmanageable. The solution, however, is still useful for a considerable time after the precipitate begins to form. In the analysis of commercial oxygen, where the residue of unabsorbed gas is very small, care must be taken that one or two of the copper wires project clear up into the entrance to the capillary; for, if the gas residue is not in contact with metallic copper, the last traces of oxygen will not be absorbed. It would appear therefore that the absorption of oxygen depends directly on the oxidation of the metallic copper, and that the function of the solution is merely to keep the metal surface clean and active. This reagent is cleaner to use and lasts longer than alkaline pyrogallate, and it is active at almost any temperature. When fresh, it leaves appreciable amounts of ammonia in the gas, for which reason the burette water should be kept slightly acid. The reagent cannot be used for the estimation of oxygen when carbon monoxide or acetylene is present in the mixture.

J. F. B.

The "Heat-Rise" Method of Testing Sulphuric Acid and Oleum. R. Curtis and F. D. Miles. (*J. Soc. Chem. Ind.*, 1920, 39, 64-65r.)—The "heat-rise," accurately standardised by the titration method, may serve as a simple and rapid test for use in plant control. For this purpose a fairly large sample, 400 grms. of acid, may be taken for the estimation, and the apparatus required consists simply

of an earthenware mug holding about a pint, two measuring cylinders, and a thermometer with a scale marked inside the stem. The standardisation is carried out with samples of oleum and sulphuric acid, the composition of which has been determined with the highest accuracy by the titration method. The results are plotted as graphs, from which the results of subsequent estimations are read off. In all cases the samples should be cooled to at least 25°C . Three types of test are described—(a) *Test for Oleum* (of not more than 25 per cent.): 200 c.c. of oleum at temperature $a^{\circ}\text{C}$. are poured quickly and with vigorous stirring into 200 c.c. of sulphuric acid at temperature $b^{\circ}\text{C}$. in the mug. The maximum temperature, $c^{\circ}\text{C}$., of the mixture is noted, and the rise is $c - \frac{1}{2}(a + b)$. The sulphuric acid should have a strength of between 92.5 and 94.5 per cent. The results are reliable within 0.5 per cent. of free trioxide or 0.1 per cent. H_2SO_4 . After mixing, the liquid should not fume. (b) *Test for Sulphuric Acid* (96 to 100 per cent.): The procedure is as above, but the sulphuric acid is poured into the oleum (20 to 25 per cent. SO_3). After mixing, the liquid should fume. If it does not fume, test again as for a weaker acid (below). Variation in the strength of the oleum, between 18 and 25 per cent., is without effect. The results are reliable within 0.3 per cent. H_2SO_4 . (c) *Test for Sulphuric Acid* (88 to 96 per cent.): 100 c.c. of the acid sample at temperature $b^{\circ}\text{C}$. are mixed with 300 c.c. of oleum (20 to 25 per cent.) at temperature $a^{\circ}\text{C}$. Rise = $c - (3a + b)/4$. After mixing, the liquid should fume. The error should be less than 0.5 per cent. H_2SO_4 . In factory work, where frequent tests are required, the method is extremely useful. The presence of large quantities of impurity affects the results, and it has been observed that oleum made from sulphuric acid recovered from T.N.T. waste acid gave an abnormally high rise of temperature. J. F. B.

Rapid Method for the Determination of Sulphur in Petroleum Oils.

A. W. Christie and C. S. Bisson. (*J. Ind. and Eng. Chem.*, 1920, 12, 171-172.)—After the combustion of 0.5 to 0.6 gm. of the sample in a calorimetric bomb with oxygen under a pressure of 30 atmospheres for the determination of the calorific value, the whole of the sulphur present in the oil has been oxidised to sulphuric acid. The contents of the bomb are washed through filter-paper into a 250 c.c. beaker, and the total acidity of the liquid is determined by titration. The solution is then made up to a definite volume such that an aliquot portion of 25 c.c. will contain between 0.2 and 1.5 mgrm. of sulphur. A portion of 25 c.c. is measured out into a 300 c.c. wide-mouthed conical flask and acidified with one drop of dilute hydrochloric acid; 10 c.c. of a solution of benzidine hydrochloride (8 grms. per litre) are added, the solution is shaken several times and allowed to stand for fifteen minutes or longer. The benzidine sulphate is collected in a Gooch crucible on an asbestos pad which has previously been treated with permanganate. The flask is rinsed three times with 5 c.c. of cold water, each portion being drained through the filter before the next is poured on. The asbestos pad and precipitate are then washed back into the original flask. One c.c. of 10 per cent. sodium hydroxide is added and the flask is warmed on the steam-bath to dissolve the benzidine sulphate. Water is then added to bring the volume up to about 100 c.c. and 5 c.c. of concentrated sulphuric acid are added. The flask is placed on the steam-bath and the solution is

titrated hot with $\frac{N}{20}$ permanganate until the red colour fades slowly. An excess of 10 c.c. of permanganate is added, and heating on the steam-bath continued for exactly ten minutes. Ten c.c. of $\frac{N}{20}$ oxalic acid are added, and as soon as the solution clears the titration is finished off with permanganate. The net consumption of permanganate is multiplied by the factor 0.041 to obtain the number of mgrms. of sulphur in the portion of original solution taken, this factor having been ascertained by the analysis of known solutions of sulphates. The results are essentially the same as those obtained by the gravimetric barium sulphate method. By using the entire rinsings of the bomb and concentrating to 25 c.c., accurate results may be obtained for oils containing only 0.1 per cent. of sulphur. J. F. B.

Estimation of Tin. J. M. Kolthoff and R. Van der Heyde. (*Pharm. Weekblad*, 1919, 56, 1466; *Chem. Zeit. Übersicht*, 1920, 44, 65).—The tin solution, the acidity (hydrochloric) of which should be about 4N, is treated with two drops of 1 per cent. antimony solution and 0.2 gram. of reduced iron, and boiled until the iron is dissolved, the mouth of the flask being meanwhile covered with a funnel. After the reduction a fragment of marble is added, so that the carbon dioxide produced will protect the stannous chloride from atmospheric oxidation, and the liquid is rapidly cooled. It is then diluted with 100 c.c. of water, free from air (prepared by adding 1 gram. of sodium hydrogen carbonate and a little hydrochloric acid to 100 c.c. of distilled water), an excess of $\frac{N}{10}$ iodine solution immediately added, and the excess titrated with sodium thiosulphate solution. The method is also applicable in the presence of considerable amounts of antimony as found in alloys. In such cases the mixture of tin and antimony is dissolved in strong sulphuric acid, the solution boiled with reduced iron and hydrochloric acid and filtered after reduction, and the filtrate treated as described. C. A. M.

Approximate Quantitative Microscopy of Pulverised Ores. W. H. Coghill and J. P. Bonardi. (*Techn. Paper*, No. 211, Bureau of Mines Dept. of Interior, U.S.A., 1919, 20 pp.).—Hynes' method of sorting pulverised minerals by the use of a reading-glass and microscope (*Min. and Sci. Press*, 1915, 110, 994) has been simplified and developed. The best apparatus for the purpose is a binocular microscope with a stage adapted for rapid orientation. The pulverised ore for each reading is oriented by adjusting it along a ruled off-set, each square of which has a distinguishing letter. Although only a part of the grains sprinkled on to the slide is counted, groups of them are taken systematically as in ordinary sampling. The most satisfactory illumination is obtained when dark-green paper is placed as a background beneath the glass slide. Close grading by means of screens is essential, and the separated products should be washed and dried so as to show clear-cut surfaces. Coarser products should be sprinkled on to the slide, so that each field contains about a dozen grains, whilst finer products should be sifted on to the slide. In preparing samples it is best to begin with wet screening on the finest sieve, and for this purpose a solution of saponin or of soap is much more satisfactory than water. In calculating the weight of constituents from the volumes counted, chemical analysis and familiarity with the ore in question will afford guidance. For example,

in the case of molybdenite concentrate the siliceous gangue has a weight factor of 1, whilst pyrites and equivalent molybdenite in "locked" grains have a factor of 2, and free molybdenite flakes (which break into thin flakes about half as thick as the other grains) are assigned a factor of 1, instead of 2, in accordance with their specific gravity. A camera lucida sketch of the outlines of the powdered mineral as seen through a monocular microscope affords a trustworthy criterion of the nature of an ore.

C. A. M.

APPARATUS, ETC.

Analytical Weighing. H. L. Wells. (*J. Amer. Chem. Soc.*, 1920, **42**, 411-419.)—The author recommends the adoption of short swings of the balance in weighing, and adduces many arguments against long swings.

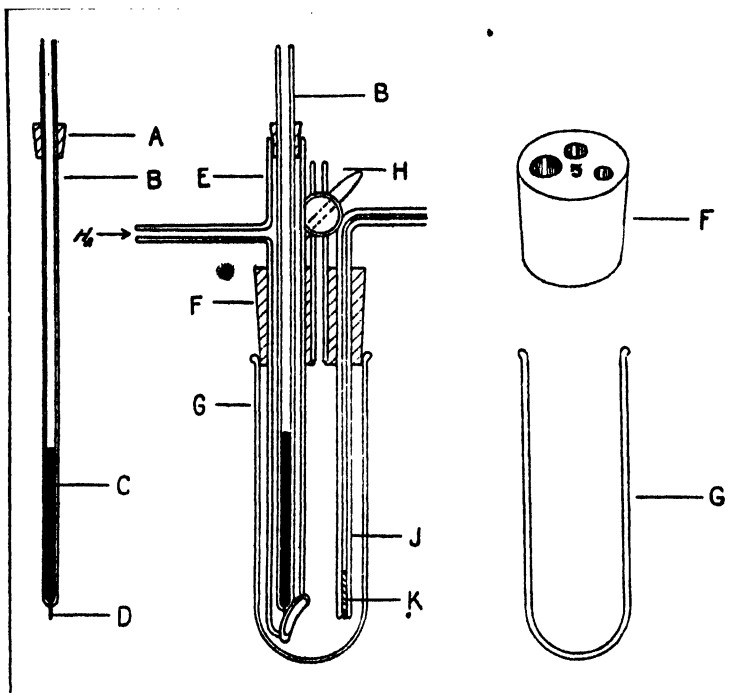
W. P. S.

Modification of the Apparatus for the Estimation of Arginine Nitrogen by Van Slyke's Method. G. E. Holm. (*J. Amer. Chem. Soc.*, 1920, **42**, 611-612.)—A Kjeldahl flask, having a short neck, is closed with a rubber stopper, through which pass a short tube for the introduction of liquids and the stem of an "anti-spray" bulb; this stem is longer than usual, and around it is fitted a short condenser. During the alkali digestion, water is passed through the condenser; the contents of the flask are then allowed to cool, the water is drained from the condenser, 200 c.c. of water and a quantity of zinc dust are added to the flask through the short tube, and the distillation is continued in the ordinary way.

W. P. S.

Determination of Hydrogen Ion Concentration. J. W. M. Bunker. (*J. Biol. Chem.*, 1920, **41**, 11-14.)—An electrode and vessel are described which fulfil the necessary requirements for easy and accurate work on the reactions of culture media and other biological fluids. The apparatus is a modification of the bubbling electrode used in a glass vessel, the essential part of the apparatus being the tube *E* used to conduct the hydrogen gas to the platinum wire of the hydrogen electrode. This is a piece of glass tubing of $\frac{5}{16}$ inch internal bore, with a side arm of small diameter joined to it at approximately 1 inch from the top, the total length being about 5 inches. In its preparation it is sealed at the bottom and then heated on one lower "corner," and a bubble at this point is blown and later broken with a file. The edges of the lip which remain are fire-polished and then the end of the tube is sealed, and the round hole thus obtained is contracted to an approximately oval shape. The hydrogen electrode itself, *B*, is made from a piece of $\frac{1}{8}$ inch tubing and a short piece of platinum wire sealed in at the bottom, projecting outside about $\frac{1}{8}$ inch, and inside far enough to make contact with the mercury *C*, the entire electrode being about 6 inches long. The tube *B* with the stopper *A* is inserted in tube *E* and adjusted so that *D* is opposite the oval orifice. When gas is bubbled through there should form a large bubble at the base of the tube *E* which is held together by the walls of the tube until it has travelled far enough for the level of the liquid within the tube to have reached a point below the platinum wire; it is thus

exposed to hydrogen gas. Electrode vessel *G* is an ordinary test-tube of 1 inch internal diameter and about 3 inches long, and when a series of determinations of P_H is to be made the proper number of tubes is selected, and one by one each may be pushed up under the electrode against the rubber stopper *F*, which has three holes



made in it to receive tightly the tube *E* and the outlet tube *H* with its stopcock, and also to receive, but not too tightly, the potassium chloride arm *J* made of small capillary tubing. Numerous working details are given, for which the original paper must be consulted.

H. F. E. H.

Problems and Fallacies of Sampling. W. L. Baillie. (*Chem. Trade J.*, 1920, 66, 333-336.)—This paper indicates the great importance of accurate sampling and the difficulties met with in its achievement. The experimental error in sampling should be of such an order that it will not increase the experimental error in the method of examination involved. Any degree of refinement is possible by taking increasing numbers of very small portions evenly from different parts of the bulk, but the probable error is inversely proportional only to the square root of the number of portions forming the sample, and this method of increasing accuracy soon becomes impracticable. When sampling materials received in numerous packages where no wilful adulteration is suspected, and the impurities consist of harmless diluents, it is probably sufficient to sample one in five packages in consignments of fifty or over, but a higher proportion is advisable in smaller lots. When, however, the impurities are liable to consist of intentional adulterants or are of an objectionable nature, the only

alternative is to sample from most or all of the packages. The importance of this latter method is illustrated by a table (quoted from Hill) showing the variation in content of arsenic and lead in different casks of a consignment of boric acid :

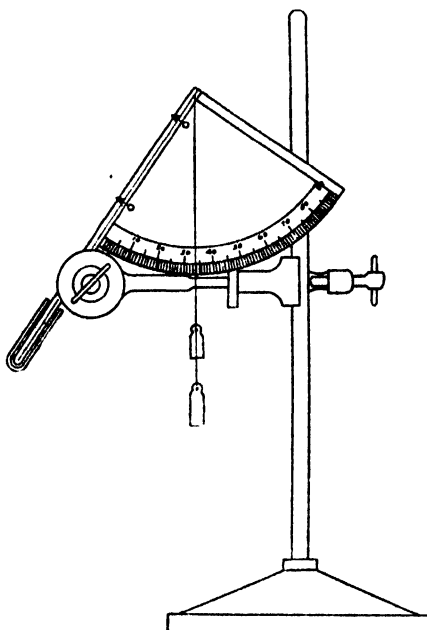
Number of Cask.	Parts per Million.	
	Arsenic.	Lead.
2	10	4
4	15	2
6	2.4	2
8	0.4	45
10	0.4	24

If the contents of any particular package differ in appearance, colour, odour, etc., from the remainder of the bulk, samples should be taken from this and each of the neighbouring units ; also a larger proportion should be taken from the first and last few packages of any delivery, particularly if the material is made by a discontinuous process. In many cases samples from different packages of a consignment may be kept separate for moisture determination, and then mixed and resampled for the remaining determinations. The amount and distribution of impurities is frequently very variable, even in a single parcel, and with some mixed materials (*e.g.*, dynamite) the composition is liable to vary owing to segregation on standing. In such cases extreme care is necessary to ensure a truly average sample. When liquids are sampled, solid foreign matter may generally be mixed uniformly by rolling or stirring, but if this is impossible a special sampling instrument must be employed. In addition to obtaining a representative sample such questions as the suitability of containers, methods of storage, means of transit, etc., must be considered. Any receptacle should be unacted upon, either physically or chemically, by the material of which the sample is composed, and should be closed in such a manner that no constituent of the sample can be lost by evaporation. With regard to labelling, this should be as complete as possible, for neglect of this precaution often leads to much unnecessary trouble and waste of time. Examples of research applied to the sampling of copper bullion and wood pulp are given, and indicate that the question of sampling is by no means a simple matter, and that an efficient sampler must possess skill and knowledge of a high degree. Several devices employed for sampling consignments of materials and for the control of manufacturing operations are described in detail, and tables illustrating various points dealt with in the text are provided.

T. J. W.

Surface Tensimeter for Small Quantities of Liquids. C. C. Kiplinger. (*J. Amer. Chem. Soc.*, 1920, **42**, 472-476.)—An apparatus for measuring surface tension which requires only a few drops of the liquid is shown in the figure. A capillary tube, 18 cm. in length and 1 mm. internal diameter, is fixed to the edge of a graduated

quadrant (this may be cut from a celluloid protractor). A short column of the liquid is introduced into the capillary, the lower end of the latter is closed with a glass cap to retard evaporation, and the tube is then turned to such an angle that the meniscus at the lower end of the liquid changes to a plane surface. This angle is given by the weighted threads attached to the apex of the quadrant, two threads being used to eliminate parallax. If A is the angle and L the length of the liquid layer in the capillary (measured to 0.1 mm.), then $L \cos A = h$, where h is the height of the



vertical column exerting the same downward pressure as the inclined layer of liquid. The formula

$$T = \frac{d \times h \times r \times 980}{2} \text{ dynes per sq. cm.}$$

is used in the calculation of T , the surface tension. Results obtained by the use of the apparatus with water, benzene, carbon tetrachloride, toluene, and ethylene dibromide are concordant but uniformly lower by a few units than the correct values; this may be due to a slight change in the length of the liquid layer owing to the flattening of the meniscus, and the need of a correction factor is indicated.

W. P. S.

Thermoregulator with the Characteristics of the Beckmann Thermometer. R. B. Harvey. (*J. Biol. Chem.*, 1920, **41**, 9-10.)—With low temperature regulation it is desirable to reduce the heat capacity of the regulator as much as possible in order to effect quick response, and it is also advisable that the thermoregulator should show the same lag effects as the standard Beckmann thermometer. An instrument is described and illustrated which can easily be set at any temperature

between -20 and $+250^{\circ}\text{C}$. The approximate setting is made by warming or cooling the mercury bulb until the upper level of the column reaches the desired point on the setting scale, while, for more accurate settings, a series of small globules of mercury is detached from either portion; as many of these droplets as are desired can be added to the contents of the bulb by turning the instrument upside down. The setting is easier than with the ordinary Beckmann thermometer, and the lower platinum contact should be made in an enlargement of the capillary to prevent sticking of the mercury at this point, the upper contact being placed at the end of the capillary so that small temperature changes will make or break the circuit. The mercury column is of sufficient diameter to carry the current desired to operate a 150 ohm relay using 2 or 4 volts. The heating circuit should be broken by a relay.

H. F. E. H.

New Turbidimeter for Solutions of Gelatin, Cellulose and Varnishes.

S. E. Sheppard. (*J. Ind. and Eng. Chem.*, 1920, 12, 167-169.)—For the measurement of the clarity of viscous solutions the most convenient method is that depending on the extinction of visible details in some standard object. In the method described, the thickness of liquid under examination and the conditions of illumination are kept constant while the visibility of the object is varied continuously. For this purpose the author employed cross gratings similar to those used by Ives for his test object for visual acuity. The essential part of the apparatus consists of two superposed opaque line gratings arranged to rotate relatively to each other about an axis perpendicular to their plane. Viewed by transmitted light at such a distance that the grating lines are below the limit of resolution, parallel dark bands are seen. When one plate is revolved over the other the separation of these bands alters continuously until a point of invisibility is reached when the bands can no longer be resolved. If the bands are viewed through a turbid solution the point of invisibility is reached with a smaller angle of rotation. The instrument comprises a source of light, consisting of a concentrated filament lamp and hood, a mirror for reading the scale, diffusing and reducing glasses, the crossed gratings with vernier and scale, a cell for holding the samples in the path of light in front of the gratings, a tube, and eyepiece. The samples are preferably placed in square-sided, 8-oz. bottles with screw covers, and, since these are not accurately made, they are immersed in the parallel glass-walled cell containing a medium such as monochloronaphthalene which has the same refractive index as the glass. Readings are first taken with clear water, and the rotation of the gratings necessary to extinguish the resolution of the bands is determined (angle = A max.). The jar containing the fluid to be tested is then placed in the cell and the rotation corresponding to extinction again determined (angle = A). A sufficiently accurate comparison is obtained by taking the percentage ratio of A to A max. as the clarity value compared with the standard liquid.

J. F. B.

REVIEWS.

ALCOHOL : ITS PRODUCTION, PROPERTIES, CHEMISTRY, AND INDUSTRIAL APPLICATIONS.

By CHARLES SIMMONDS, B.Sc. London: Macmillan and Co. 1919. Price 21s. net.

It does not always fall to the lot of an author to produce a scientific treatise at a time so peculiarly opportune as that selected by Mr. Simmonds. The conviction of the sub-committee on motor fuel that the only ultimate solution of the motor-fuel problem is the production of home or empire-produced power alcohol, the recent appointment of Sir Frederick Nathan as Power Alcohol Investigation Officer under the Fuel Research Board, and the necessarily increasing quantity of alcohol which is required for our expanding manufacture of dyes, synthetic drugs, etc., is ample testimony to the national importance of the production of alcohol.

Mr. Simmonds' work is of a standard character, and its compilation shows internal evidence not only of great care and accuracy, but also of scholarly enthusiasm for his subject. The arrangement is admirable, and a judicious balance is maintained of the relative importance of the several matters dealt with. The reviewer is tempted to take his readers for a mental tour through the different chapters, but he must be content with drawing attention to some of the more important aspects of the author's work. A brief introductory chapter of an historical character is followed by one dealing with the production of alcohol. A good account is given of the "Amylo" process of fermentation, and in this connection it is greatly to be deplored that our Customs and Excise regulations prevent advantage being taken of any benefits which the process may offer, or of any developments to which it may give rise. Technical progress is impeded because of the statutory requirement that the distillers' wort before fermentation shall be ascertained by means of the saccharometer, and this, unfortunately, is impracticable in the "Amylo process." A better instance of bureaucratic short-sightedness would be hard to find. The production of alcohol from wood is described, and although the results so far have not been financially successful, yet in the opinion of some technologists there is a future for the Classen process. The fact that several millions of gallons of alcohol have been produced from wood waste lends support to the view that ultimate success is probable. The main difficulties appear to be of an economical character.

The manufacture of methyl alcohol is described, and useful tables are given of densities of mixtures of methyl alcohol and water.

The analytical side, as might be anticipated, has been dealt with by Mr. Simmonds in a most lucid manner, and for this—if for no other—reason the volume will find a place in the book-shelves of those practitioners who are interested in alcoholometry and the analysis of alcohol, acetone, spirits, wines, etc. The position of industrial alcohol, the composition of denatured and specially denatured alcohols, foreign denaturants, and the various official regulations attaching to their use, are discussed. The preparation of ether, chloroform, ethyl chloride, bromide and iodide, chloral ethyl acetate, ethyl acetoacetate, and other technical ethyl and methyl compounds, is described.

The matter dealing with alcohol as a source of motive power and the author's

résumé of the most recent reports and investigations on this subject will be read with interest by those who are concerned with this far-reaching and important problem.

That portion of the book dealing with the chemical examination of potable spirits and the advantages and defects of the Allen-Marquardt, Beckmann, Government Laboratory, and other methods of estimating higher alcohols will be read with professional interest by the analyst. The analysis of beer, cider, perry, and wines is gone into in a fairly comprehensive manner. Included in the first mentioned is the refractometric determination of original gravity, which constitutes a useful brewery control method.

The final chapter, dealing with the physiological effects of alcohol, is based on the valuable review recently compiled by the Central Control Board (Liquor Traffic) of the United Kingdom.

The author is to be heartily congratulated on the production of a volume which is a real contribution to a highly important branch of chemical literature.

EDITOR.

A TREATISE ON QUALITATIVE ANALYSIS. By F. CLOWES and J. B. COLEMAN. Ninth edition. Pp. 416. London: J. and A. Churchill, 1920. Price 12s. 6d. net.

The new edition of this well-known work on qualitative analysis can be strongly recommended as a book which may safely be put into the hands of all students engaged in the study of this important subject.

In the training of those who attend the laboratories of our schools and colleges, it is most desirable that the qualities of accuracy, carefulness, and attention to detail should be inculcated from the beginning, and the teacher of practical chemistry will find that this text-book provides valuable assistance in the task of instructing those placed in his charge.

This edition is enlarged and revised. The section on the preparation of gases which appeared in former editions has been deleted, and this has allowed space for a fuller treatment of matters relating to analytical practice. A further improvement is the arrangement of the reactions of the metals so that they are now placed in their natural order, and formulæ, such as KHO, have been rewritten KOH etc.

In the first two sections, apparatus and general manipulation are thoroughly treated in a manner calculated to induce in the student a respect for *method* and clean working. A better diagram of the Bunsen flame should be given, showing its various parts, and instructions for cutting glass tubing of wide bore might be included.

In a book which claims to be a *treatise*, a more up-to-date account should be given of the spectroscope and of the various methods of illumination employed in working with it, as well as a description of the means employed for ascertaining the wave-lengths of the spectrum lines. The treatment of the subject is too elementary.

In the section dealing with analytical reactions, the theoretical side of the subject has received far less attention than it merits. Surely, the scientific foundations of analytical chemistry give spirit and life to the subject, and are calculated to stimulate enthusiasm. No mere consideration of *space available* should allow this aspect of the subject to be almost ignored.

The two following sections deal with the reactions of the metals and acid radicles. The explanations are clear and concise, and the treatment thorough. The reactions of the rarer elements are included, together with tables by means of which the various metals and acid radicles may be separated and identified. In this connection it would be a great advantage if, in presenting two or three alternative schemes of separation, the authors would give us the benefit of their experience, and state which scheme is advisable under any given set of conditions.

There is no mention of the following reactions which are now extensively employed, and which should be given in a book of this scope—namely, cupferron for the precipitation of iron and titanium, the hydrogen peroxide test for titanium, the dimethyl-glyoxime test for nickel, and the α -nitroso- β -naphthol test for cobalt; also the ammonium cobalto-thiocyanate test for this last metal.

Further omissions are: the sulphanilic acid and α -naphthylamine test for nitrites, the perchloric acid test for potassium, the sodium cobalti-nitrite test for that metal, and the pyroantimoniate test for sodium. It is not made sufficiently clear that lithium carbonate is not precipitated in the barium group in the presence of much ammonium chloride, and the formula for ammonium metavanadate is wrongly written.

There is a useful section dealing with the reactions of the more common organic compounds, and on the identification of gases; the portion dealing with the analysis of complex mixtures, minerals and acid radicles is particularly good, and includes ample explanations of numerous important points.

The tables given for the separation of the rarer metals need some extension, in particular, that dealing with the analysis of rare earth minerals.

Sound advice is given concerning laboratory fittings and arrangements, and the expression of the concentration of reagents in terms of normality is rightly advocated.

The book is full of useful information, and the numerous cross-references have permitted the inclusion of much matter within a small compass. It will no doubt find increasing favour among teachers and analysts.

The authors are evidently exponents of the gospel of *orderliness* and the *clean bench*, factors so essential to all good work. The spirit of the book is an antidote against haphazard and "slapdash" work which so easily spoils an otherwise promising student, and the authors are to be congratulated upon providing us with such an excellent treatise on the subject.

A. J. HALE.

ANNUAIRE POUR L'AN, 1920. Paris: Gauthier-Villars et Cie. Price 4 fr. 50 c.

This little book is published under the ægis of the Bureau des Longitudes, and appears to have been issued regularly since 1796.

It contains an immense mass of useful information in the fields of astronomy, meteorology, geography, chemistry, and physics. Among the information likely to be useful to the chemist, one notices the numerous tables giving the densities of pure substances and of minerals, co-efficients of expansion, vapour tensions, specific heats, indices of refraction, specific rotatory powers, and many other chemical and physical data.

There are also brief but clearly written explanatory chapters dealing with such subjects as alcoholometry, radio-activity, calorimetry, etc.

The compilers and the Bureau des Longitudes are to be congratulated on the evidence of careful selection which this little book reveals, and on having compressed such a vast amount of useful matter within so small a compass. As a "pocket-book" of general reference it is one of the best which has come under the writer's notice.

A. CHASTON CHAPMAN.

CATALYSIS IN THEORY AND PRACTICE. By E. K. RIDEAL and H. S. TAYLOR. Pp. 496. London: Macmillan and Co. 1919. Price 17s. net.

The great interest of catalysis to chemists in general rests chiefly on two facts. First, catalytic action is so general that it is questionable whether any reaction has ever been conducted without catalysis, and, second, such explanations as have been advanced are obviously primitive and tentative, and afford little assistance to the chemist who is in search of the most suitable catalyst for some particular reaction. Probably no greater service could be rendered to the science, both theoretical and applied, than the formulation of a satisfactory general theory, or set of theories, to explain catalytic action. If and when this is accomplished, chemical action in general will doubtless be understood in a manner altogether more fundamental than is the case to-day.

If the reader of the volume under review expects to find in it new light on catalytic processes, he will be disappointed. The work is purely a compilation from previous publications, with occasional notes from the authors' own experience and, here and there, remarks on possible directions for future research. The discussion of theory is slight and uncritical. It follows, on the whole, the Ostwald dogma.

Regarded as a compilation, the work has much in its favour. It is comprehensive, as the following titles of its chapters indicate: Early History; Theoretical Introduction; Measurement of Reaction Velocity in Catalytic Processes; Oxidation Processes; Hydrogen and Hydrogenation; Dehydrogenation; Fixation of Nitrogen; Hydration and Hydrolysis; Dehydration; Applications of Catalysis to Organic Chemistry; Ferments and Enzymes; Catalysis in Electrochemistry; Catalysis by Radiant Energy; Catalysis in Analytical Chemistry.

The analytical chemist will probably turn first to the last chapter, of some forty-three pages, as embodying his immediate interests. It is doubtful whether he will find anything of novelty in it, but if he is not in touch with other developments of the science he may obtain hints from other sections of the work which will enable him to choose a suitable catalyst for some reaction which he desires to accelerate. In the present state of the theory there can be only occasionally any certain guide in this matter.

The book is pleasingly printed, but with rather more "printer's errors" than should have escaped correction. The references are numerous, but at times seem to have been overlooked—*e.g.* pp. 179-182. In other ways also there is evidence that the production has been hurried—*e.g.* the reader is supplied with many chemical equations which he could be trusted to know (*e.g.* the one on p. 139), while the less

familiar formulæ of mathematical physics are employed with no, or little, explanation. These minor faults may well be corrected in a second edition.

H. F. COWARD.

THE PROFESSION OF CHEMISTRY. By RICHARD B. PILCHER. Pp. xiv and 199. London: Constable and Co., Ltd. 1919. Price 6s. 6d. net.

The choice of a career for a boy or girl is generally a difficult matter, and especially so when those with whom the choice lies are unacquainted with the means and qualifications necessary to success in the career that may be contemplated. No one perhaps is better qualified than Mr. Pilcher to advise as to training for the chemical profession, and his book bears the impress of thorough knowledge of his subject. The book will prove extremely helpful to parents and guardians who have this profession in view for their charges, as well as to the youthful aspirants themselves. Many who entered the profession years ago have since had occasion to regret that the advice contained in this book was not available when it could have been of use to them.

Mr. Pilcher takes a comprehensive view of his subject, beginning with general education at school, passing thence to college or university training, and finally outlining the special qualifications requisite in the various branches of the profession. Naturally and properly much space is devoted to the requirements of the Institute of Chemistry. The section on "Pharmacists and Chemists" will be especially instructive for those for whom the book is primarily intended; a good case is made out for the claim to the title "Chemist" of those who have studied the science generally, as distinct from those who have studied the one branch of applied chemistry known as pharmacy. The section on "Professional Training" includes a useful list of Institutions where special courses in various branches of applied chemistry may be obtained. In the section on "Professional Procedure" Mr. Pilcher upholds a high standard of conduct, and in dealing with the subject of advertising he quotes very aptly an opinion of Dr. Johnson, who, whilst condemning solicitation for employment by a lawyer, said: "I would have him inject a little hint now and then to prevent his being overlooked." The "little hint" must be allowed to chemists as well as to lawyers, though of course much depends on the manner of application. The book concludes with a short account of the services rendered by chemists during the late war. In reference to the Gas Services it is remarked that "High qualifications were unnecessary for the work of the rank and file," a statement that will be contradicted by none.

Mr. Pilcher does not hold out illusory hopes of riches to those who intend to enter the profession, and indeed the impression left on the mind of the reader is that, whilst the study of chemistry demands high thinking, the practice of it enforces rather plain living. That this impression is correct will be generally admitted. It is unfortunately the case that the man who has spent some of the best years of his life in training to become a chemist often finds his services less well rewarded than those of the man who has passed direct from school to commercial work. It may be that this condition is one of unstable equilibrium, and that time will effect an

improvement. The attitude adopted by Government and Municipal authorities towards the profession and towards science generally is largely responsible for the present unsatisfactory state of affairs. It is to be hoped that the Government will profit by the lesson of the war in this respect and learn wisdom from our late opponents.

L. EYNON.

INTRODUCTION TO PHYSICAL CHEMISTRY. By JAMES WALKER, LL.D., F.R.S.
Eighth Edition. Pp. xiii + 433. London: Macmillan and Co., Ltd. 1920.
Price 16s. net.

The first edition of this excellent book appeared twenty years ago, when the English-reading student of chemistry was almost entirely dependent on translations of German textbooks of physical chemistry. It was at once widely adopted as a textbook in our universities and colleges, and, from the fact that an eighth edition has recently been called for, it is clear that the work is still highly esteemed by teachers and by students.

It is unnecessary to describe in detail the plan of a work so familiar to all students of chemistry as that under review. In the eighth edition the general plan of the first is still found, but the necessary revision has, of course, been carried out for the purpose of bringing the information up to date, and new material also has been introduced. We have then before us a perfectly modern, lucidly written elementary textbook of physical chemistry, in which the student will find the fundamental facts and theories of physical chemistry clearly set out, and their importance in the older branches of the subject indicated.

At the time when the book under review was first published, the author felt the necessity of effecting a connection between the ordinary chemical knowledge which the student was supposed to possess and the new material forming the content of physical chemistry. By the method of treatment adopted by the author it was hoped to prevent the student from shutting up his knowledge of physical chemistry in a water-tight compartment, and so failing to perceive its connection with and importance for the proper understanding of the chemistry of inorganic and organic substances. One would hope that the same necessity does not exist to-day. The older manner of teaching inorganic chemistry has, with the growing up of a new generation of teachers, largely disappeared, and many of the more important facts and theories of physical chemistry are now introduced in the appropriate manner even in the elementary courses in inorganic chemistry. The teaching of organic chemistry, it is to be hoped, will similarly undergo changes under the influence of physical chemistry. Although, therefore, the necessity for emphasising the organic connection between physical chemistry and the older branches of the subject may not be so great as formerly, the work by Professor Walker will be found of the greatest value to the teacher who desires to keep the unity of the subject constantly before the minds of his students.

In the present edition two new chapters make their appearance, dealing with the dimensions of atoms and molecules, and with atomic number. In reading through these chapters dealing with such important matters, the reviewer could not

but feel that the author had carried the process of condensation rather far, and he would venture to suggest that a somewhat fuller treatment might be given to these subjects in a future edition.

To those familiar with Professor Walker's book it is only necessary to say that the present edition preserves the high standard and good qualities of the earlier editions; while to those who have not yet become acquainted with the work, the warmest recommendation can be given.

A. FINDLAY.

MINISTRY OF HEALTH.

EFFECTIVE CONTROL OF CERTAIN THERAPEUTIC SUBSTANCES.

THE MINISTER OF HEALTH has appointed a Committee to consider and advise upon the legislative and administrative measures to be taken for the effective control of the quality and authenticity of such therapeutic substances offered for sale to the public as cannot be tested adequately by direct chemical means.

The Committee consists of the following members :

Sir Mackenzie D. Chalmers, K.C.B., C.S.I. (Chairman).

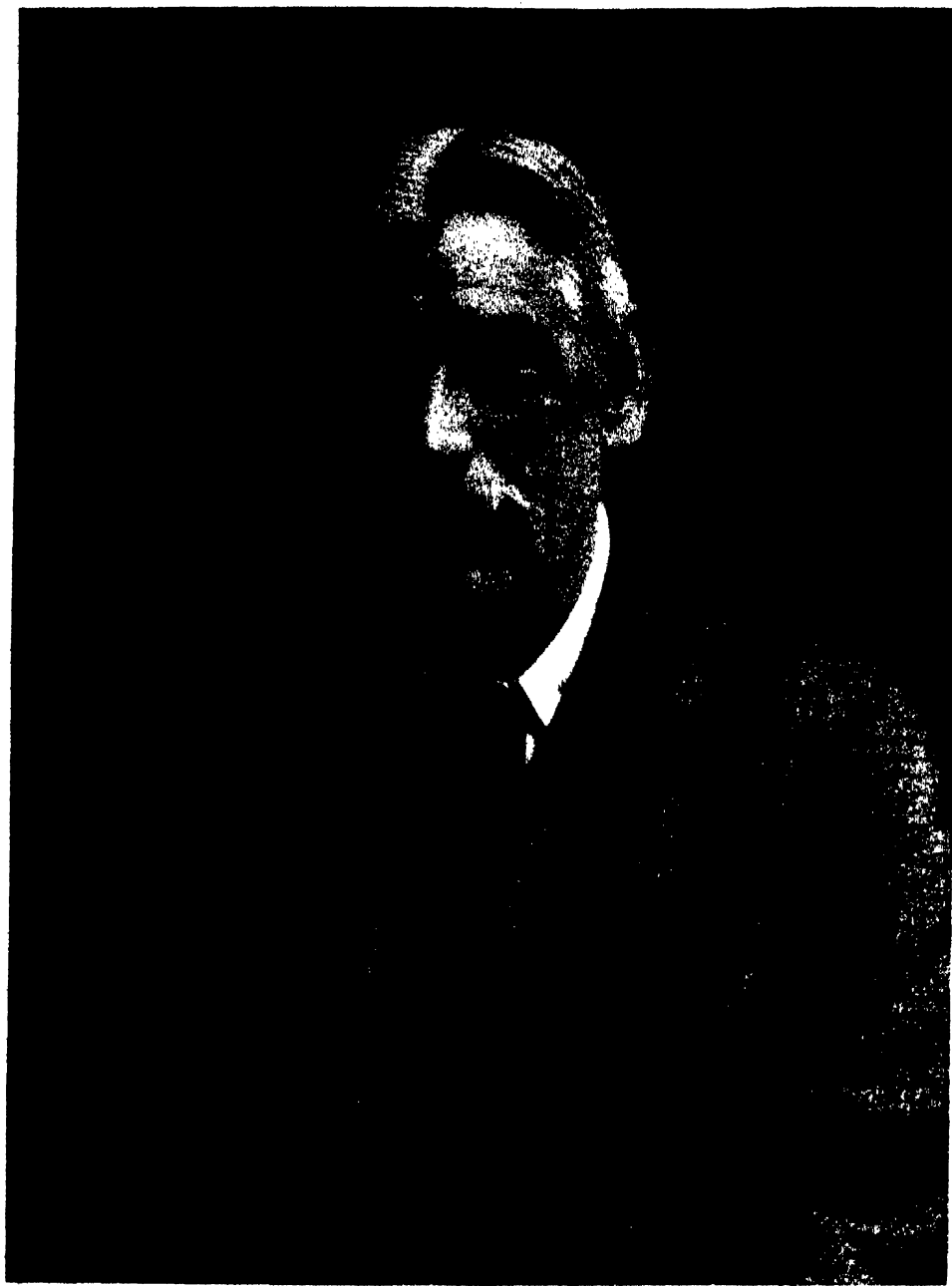
H. H. Dale, Esq., C.B.E., M.D., F.R.S., Head of Department of Biochemistry and Pharmacology under Medical Research Council.

G. F. McCleary, Esq., M.D., Medical Officer, Ministry of Health.

A. B. MacLachlan, Esq., Assistant Secretary, Ministry of Health.

C. J. Martin, Esq., C.M.G., D.Sc., F.R.C.P., F.R.S., Director, Lister Institute of Preventive Medicine.

The Secretary of the Committee is E. W. Adams, Esq., O.B.E., M.D., of the Ministry of Health, Whitehall, S.W. 1.



7. Swam. Ostend. photograph.

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W. W. Fisher

THE ANALYST.

OBITUARY.

WALTER WILLIAM FISHER, M.A., F.I.C.

THE late Walter William Fisher, M.A., F.I.C., Aldrichian Demonstrator of Chemistry in the University of Oxford, who died on February 7, in his 78th year, was one of the oldest members of the Society of Public Analysts, and acted as its President in 1889 and 1890. He was Public Analyst for the counties of Oxford, Berks and Bucks, and also for the city of Oxford, having held these posts for nearly forty years, and he was actively engaged in the duties appertaining to them up to within a week of his death.

He was educated at Worcester and Merton Colleges, Oxford, took a first-class in Chemistry in the School of Natural Science in 1870, and, with the versatility which characterised him, proceeded the next year to take a class in "Greats." He was shortly afterwards elected a Fellow of Corpus Christi College. His classical study was but an interlude, for he at once returned to his chemical work as assistant to the late Prof. Vernon Harcourt, and in the following year was appointed by Sir Benjamin Brodie as University Aldrichian Demonstrator in Chemistry, which post he held until his death. He was for six years, from 1874, lecturer in Chemistry at Balliol, and he acted on various occasions as Public Examiner for the University. He also acted as Examiner to the Institute of Chemistry from 1903 to 1907, and was a Member of Council of that body in 1893-1896, 1898-1901, and 1907-1910.

The transactions of the Chemical Society include papers by Fisher on "Manganese Tetrachloride" (1878) and "Lead Tetrachloride" (1878), while a paper in the "Proceedings" of the same Society appeared in 1892 on "Anhydrous Oxalic Acid." To the "Proceedings" of the Society of Public Analysts he contributed papers on "Alkaline Waters from the Chalk" (ANALYST, 1901), and "Alkaline Waters from the Lower Greensand" and "Indirect Estimation of Alkalies in Waters" (ANALYST, 1902). Six or seven years prior to his death he had been engaged in the compilation of a paper on "Some Variations of the Thames Water," which he hoped to read before the Society. He was a good geologist, and made a special study of the composition of well waters derived from various water-bearing strata, apart from the publication of the papers to which reference has been made. He was also all his life a keen field botanist.

During his Presidency of the Society of Public Analysts he gave valuable evidence on behalf of the Society before the Departmental Committee which was

appointed in 1899 by the President of the Local Government Board to inquire into the use of preservatives and colouring matters in foodstuffs.

Fisher's versatility was not confined to intellectual matters. He joined the Volunteers in 1860, and was a member of the 1st Surrey Rifles for eleven years, during which time he won many prizes for shooting; and at college he rowed in the Merton eight, and was the possessor of many cups for various branches of athletics.

Personally, he possessed a charming manner which faithfully expressed his gentle and cultured spirit, and he was a loyal and affectionate friend beloved by his colleagues and by many generations of pupils.

BERNARD DYER.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

ORDINARY MEETING, MAY 5, 1920.

HELD at the Chemical Society's Rooms, Burlington House, Mr. Alfred Smetham, President, in the chair.

A certificate was read for the first time in favour of Mr. Udolphus Aylmer Coates.

Certificates were read for the second time in favour of Misses Dorothy Gertrude Hewer, B.Sc. (London), Rita Catherine Hawkins Johnson, Messrs. Harold Hall, F.I.C., Geoffrey Trelawney Bray, A.I.C., Frank William George King, John Herbert Stubbs, M.Sc. (Victoria), F.I.C.

The following were elected Members of the Society: Messrs. Laurence Harry Mills, B.Sc. (Birmingham), A.I.C., Frederick Robertson Dodd, F.I.C.

The following papers were read: "Estimation of the Age of Ink in Writing," by C. Ainsworth Mitchell, M.A., F.I.C.; "Examination of Crude Chinese Camphor," by E. R. Dovey, A.R.C.S., A.I.C.; "A Volumetric Method of Estimating Iron," by H. Droop Richmond, F.I.C., and Edith M. Ison, B.Sc. (London).

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AN EXAMINATION OF CERTAIN MILK RECORDS.

By G. W. MONIER-WILLIAMS, M.A., PH.D., F.I.C.

(Read at the Meeting, April 14, 1920.)

THIS paper deals with the results of the examination of certain series of milk records which have been placed at my disposal at various times during the last eight or ten years.

These records have been submitted to elementary statistical analysis, and the results, although far from complete, are, I think, of sufficient interest and importance to show the value of this method of dealing with milk records. I should mention that I have not in all cases been in a position to test the reliability of the actual analytical data, but have assured myself that the work has been conducted by skilled operators. All the fat analyses have, I believe, been carried out by the Gerber method. Although slight inaccuracies may possibly have arisen in some cases owing to the use of uncalibrated apparatus, and from other causes, they would not be sufficient to affect the broad conclusions which may be drawn from the results.

The first series is one of 4,825 samples of milk from single cows of one herd, taken over a short period. The experiments were carried out under the direction of Mr. Wilfred Buckley, Director of Milk Supplies, on his own herd at Moundsmere Manor, near Basingstoke, in 1915, and I am much indebted to him for placing the results at my disposal and allowing me to bring them before this Society.

The herd consisted of sixty-five dairy Shorthorn cows, counting only those which were in milk at the time. The milk of each cow, both morning and evening, was weighed, and the fat content determined, over a period of thirty-nine days—from April 18 to May 26, 1915. During the first part of this period, up to May 5, the cows were on winter rations, and were turned out during the day in a 2-acre paddock. During the last half of the period they were turned out to grass, and no concentrated food of any kind was given.

For the purpose of the experiments the cows were divided into three groups, according to the stages of the lactation period :

Group I.—Twenty-four cows which had calved from January 9 to March 25 of the year in which the records were taken.

Group II.—Twenty cows which had calved from October 15 to December 27 of the previous year.

Group III.—Twenty-one cows which had calved from June 1 to September 18 of the previous year.

As the period covered by the experiments advanced, several of the cows in Group III. dried off, so that at the end of the thirty-nine days only fourteen cows out of twenty-one in this group were giving milk.

The results obtained are shown in Table I. :

The actual percentages of samples falling below 3 per cent. of fat were as shown in Table II., while Table III. shows the way in which these samples deficient in fat were distributed throughout the herd.

TABLE I.

				Number of Samples.	Average Fat Content per Sample.	Average Daily Yield per Cow.	
						Milk in Pints.	Milk Fat in Pounds.
1. Morning Milk.							
(i.) <i>Separate groups :</i>							
(a) Winter rations—							
Group I.	...	...	430	2·87	13·6	0·50	
Group II.	...	...	359	3·24	11·4	0·48	
Group III.	...	...	348	3·66	5·6	0·26	
(b) Grass fed—							
Group I.	...	...	502	2·87	14·4	0·53	
Group II.	...	...	414	3·20	12·2	0·50	
Group III.	...	...	361	3·36	6·4	0·27	
(ii.) <i>Complete herd :</i>							
(a) Winter rations	...	...	1,137	3·23	10·5	0·43	
(b) Grass fed...	...	...	1,277	3·12	11·4	0·45	
(c) Whole period	...	...	2,414	3·17	11·0	0·44	
2. Evening Milk.							
(i.) <i>Separate groups :</i>							
(a) Winter rations—							
Group I.	...	...	432	4·17	9·1	0·49	
Group II.	...	...	360	3·98	7·5	0·39	
Group III.	...	...	348	4·46	3·6	0·21	
(b) Grass fed—							
Group I.	...	...	496	4·06	9·6	0·50	
Group II.	...	...	414	3·98	7·3	0·37	
Group III.	...	...	361	4·32	4·1	0·22	
(ii.) <i>Complete herd :</i>							
(a) Winter rations	...	...	1,140	4·20	6·9	0·37	
(b) Grass fed...	...	...	1,271	4·11	7·3	0·39	
(c) Whole period	...	...	2,411	4·15	7·1	0·38	
3. Morning and Evening Milk together.							
(i.) <i>Separate groups :</i>							
(a) Winter rations—							
Group I.	...	...	862	3·52	22·6	0·99	
Group II.	...	...	719	3·61	18·9	0·86	
Group III.	...	...	696	4·06	9·2	0·46	
(b) Grass fed—							
Group I.	...	...	998	3·46	24·0	1·03	
Group II.	...	...	828	3·59	19·5	0·88	
Group III.	...	...	722	3·84	10·5	0·49	
(ii.) <i>Complete herd :</i>							
(a) Winter rations	...	...	2,277	3·71	17·3	0·80	
(b) Grass fed...	...	...	2,548	3·61	18·7	0·84	
(c) Whole period	...	...	4,825	3·66	18·1	0·83	

TABLE II.—PERCENTAGE OF SAMPLES FALLING BELOW 3 PER CENT. OF FAT.

	Winter Ration Period.	Grass Fed Period.	Whole Period.
<i>Morning Milk :</i>			
Group I.	55.8 per cent.	57.6 per cent.	35.8 per cent.
Group II.	23.4 "	30.7 "	
Group III.	9.8 "	24.9 "	
Complete herd	31.6 "	39.6 "	
<i>Evening Milk :</i>			
Group I.	1.6 per cent.	4.6 per cent.	2.16 per cent.
Group II.	1.4 "	2.2 "	
Group III.	0.8 "	1.7 "	
Complete herd	1.2 "	3.0 "	

TABLE III.—DISTRIBUTION OF SAMPLES DEFICIENT IN FAT (MORNING MILK).

Milk fell below 3 per Cent. Fat on—	Number of Cows of—			Whole Herd.
	Group I.	Group II.	Group III.	
Nil occasions	1	1	6	8
1- 4 "	1	6	6	13
5- 9 "	2	3	4	9
10-19 "	4	7	4	15
20-29 "	9	2	1	12
30-37 "	7	1	—	8
				65 cows

TABLE IV.—DAILY MIXED MORNING MILK OF WHOLE HERD.

Per Cent. of Fat.	Number of Occasions.
2.79	1
2.89	1
2.93	1
2.95	1
2.96	1
2.97	1
2.98	1
2.99	2
3.00	4
3.01-3.09	9
3.10-3.19	11
3.20-3.29	3
3.30-3.39	2
3.50	1
	39

Table IV. shows the composition of the daily mixed morning milk of the whole herd, calculated from the fat percentage and yield given by each cow.

It will be seen from these results that :

1. The fat content of the morning milk is remarkably low throughout, especially in the case of cows which had recently calved.

2. The evening milk is, on the average, very rich in fat, the mean for the whole period being 4·15 per cent.

3. The sudden change from winter rations to grass has not had so great an effect as we might have expected, although the influence of the change can be seen clearly.

4. The rise in quality and corresponding fall in quantity as the lactation period progresses is very marked. The relative decrease in quantity is much greater than the relative increase in fat percentage, a relation which is reflected in the total amounts of milk-fat yielded at different stages of lactation. It has been pointed out by Crowther in 1905, and by Eckles and Shaw in 1913, that the actual rates of increase in milk-fat and decrease in yield are by no means even or gradual, but fluctuate considerably at different stages of the lactation period and under different conditions. But, for the purposes of this paper, it is sufficient if we take it that the general tendency is for the milk to become richer and for the yield to fall off as lactation progresses.

While the rise in fat percentage with progressing lactation is very evident in the morning's milk, it is not so marked in the evening's milk, in which the fat is higher during the first three months of lactation than during the middle period.

We can now compare these averages with those of the Aylesbury Dairy Company, published by Richmond.

By interpolation from Richmond's figures we can get the probable figures for Aylesbury dairy milk for the period April 18 to May 26. The comparison is shown in Table V. :

TABLE V.

	Morning.	Evening.	Mean.
	Per Cent. Fat.	Per Cent. Fat.	Per Cent. Fat.
Aylesbury Dairy Company :			
Averages for period 1906-1914	3·34	3·75	3·54
Aylesbury Dairy Company :			
Averages for 1915	3·50	3·71	3·60
Moundsmere herd :			
Averages of single cow samples	3·17	4·15	3·66
Moundsmere herd :			
Averages of mixed milk of herd	3·08	4·11	3·59

The figures on the lowest line are arrived at from the following considerations : The comparison of averages of single cow milks with those of mixed milks is not strictly permissible, by reason of the variation in the yield of milk from cows in different stages of lactation.

From Table I. it is evident that a low fat percentage corresponds in most cases to a high yield of milk. On mixing the milk, the fat content of the mixed milk will be affected to a greater extent by the more bulky samples, thus bringing the average fat content down. If we have, for instance, two samples, one of 6 quarts at 3 per cent. of fat, and the other of 2 quarts at 4 per cent. of fat, the average fat content of the samples will be 3.5 per cent., but, on mixing the two samples, the fat in the mixed milk will only be 3.25 per cent. I have therefore calculated from the fat percentage and milk yield in each case what would be the daily composition of the mixed milk of the whole herd, and then taken the averages from the figures thus arrived at. These averages are, I think, fairly comparable with Richmond's figures.

It would appear therefore that, judged by the final average, the milk of this herd is in the aggregate normal, and that it is the distribution of milk-fat which is at fault. At the same time, the actual quantity of milk-fat yielded by the Moundsmere herd is probably somewhat less than the Aylesbury Dairy Company average, owing to the difference in the quantity of morning and evening milk. If we take it that the yield of morning milk is 50 per cent. greater than that of the evening in both cases, there is approximately a difference of 3 per cent. in the total yield of milk-fat in favour of the Aylesbury Dairy milk—that is to say, if the average daily yield of milk-fat calculated from the Aylesbury figures is taken as 100, that of the Moundsmere herd is 97.

One of the prime causes of inequality in distribution of fat is admitted to be unevenness in intervals between milking. In this herd the milking intervals were approximately nine and fifteen hours—that is to say, the cows were milked at about 5 a.m. and again at 2 p.m. for the greater part of the period. Whether this is sufficient to account for the uneven distribution of fat is doubtful. Table VI. shows the effect of changing the milking intervals during the period when the herd was out to grass.

The herd had been divided for milking purposes into three squads, X, Y, and Z, each of these squads comprising an approximately equal number of animals at varying stages of lactation. On May 20 the order of milking the squads was reversed, so that in Squad X the milking intervals were 7 and 17 hours, and in Squad Z 11 and 13 hours approximately, Squad Y remaining the same as a control.

TABLE VI.

Period				Milking Intervals.	Morning Milk.	Evening Milk.
					Per Cent. Fat.	Per Cent. Fat.
X Squad	1st week	...	...	9 and 15 hours	3.22	4.19
	2nd week	...	...	7 and 17 hours	3.09	4.44
Y Squad	1st week	...	...	9 and 15 hours	3.02	4.19
	2nd week	...	...	9 and 15 hours	3.02	4.14
Z Squad	1st week	...	...	9 and 15 hours	3.09	4.34
	2nd week	...	...	11 and 13 hours	3.03	3.79

The changes brought about by the alteration in milking hours are in the direction of what we should expect, with the exception of the morning's milk of Squad Z, which remained low, notwithstanding the equalisation of the milking intervals. This may indicate that some other factor was at work in causing the morning's milk to be so low, but possibly a week may have been too short for the cows to accustom themselves to the changed conditions.

I think that fresh light can be thrown on these and other milk records by a study of the so-called "frequency" curves. If a number of results, which vary normally about a mean value, are plotted out so as to show the percentage of results falling at the various milk-fat percentages, a frequency curve is obtained of the general type shown in the dotted lines in the figures. The greatest number of samples cluster round the mean and on both sides of it, the number showing variation from the mean becoming progressively smaller as the extent of the variation increases. It is obvious that the greater the "dispersion" of the results—that is, the more variable they are—the flatter will be the curve.

It does not by any means follow that all series of experimental results conform to the normal law of variation and give a regular and symmetrical curve; in fact, the curves resulting from most measurements of naturally occurring variations show what is termed a definite degree of "skewness"—that is to say, the "mode," or point at which the greatest number of results fall, is shifted more or less to one side of the mean.

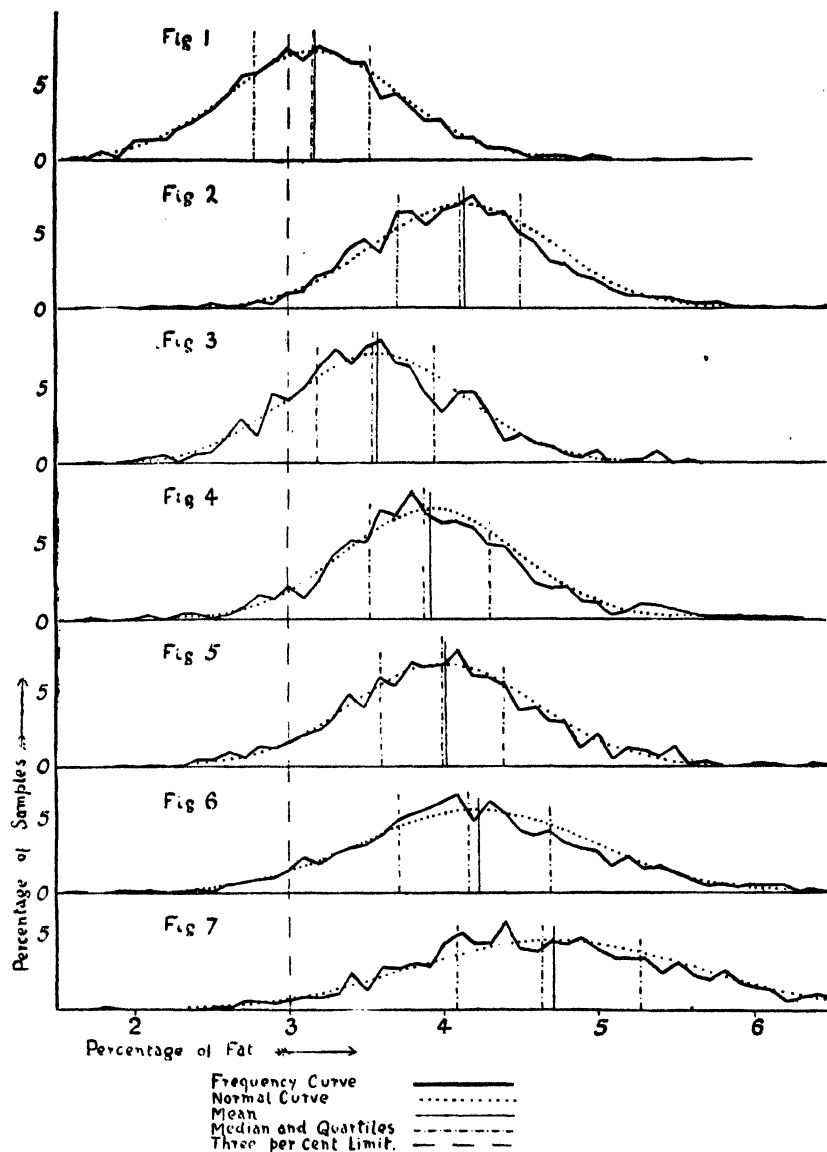
A normal frequency curve is characterised by the so-called "probable error," or, to use a term more suited to the present purpose, the "probable variation." This may be roughly defined as that degree of variation from the mean which includes 25 per cent. of the results on either side, or 50 per cent. in all.

The "probable variation" is, therefore, a simple way of expressing numerically the relative dispersion of all the results.

Several other terms have been introduced into the nomenclature of frequency curves, such as "coefficient of variability," "standard deviation," etc., but as these are mostly derived mathematically by means of the method of least squares, it is perhaps clearer and simpler to refer only to the "probable variation." In all these curves the "probable variation" has been ascertained as follows:

The exact positions of the "median," or middle point, of all the results and of the "quartiles," or quarter and three-quarter points, have been ascertained by arranging the results in groups and by interpolation. The group interval in each case is 0.1 per cent. of fat. The distance between the two quartiles, represented by the vertical dot and dash lines in the diagrams, may be termed the "50 per cent. zone." Half of this is taken as the "probable variation."

For the value thus obtained for the "probable variation" the normal curve has been drawn, using data which are to be found in mathematical tables. The normal curve is fitted to the results so that the "means" of the two curves coincide. It is useful to give these details, as there is more than one method of arriving at the probable error or probable variation, and also of fitting the normal curve to the frequency curve.



EXPLANATION OF CURVES.

- FIG. 1.—Moundsmere herd morning milk from April 18 to May 26, 1915. 2,414 samples from single cows. Probable variation, 0.380.
- FIG. 2.—Moundsmere herd evening milk. 2,411 samples from single cows. Probable variation, 0.396.
- FIG. 3.—Lanark morning milk. 754 samples from single cows. Probable variation, 0.380.

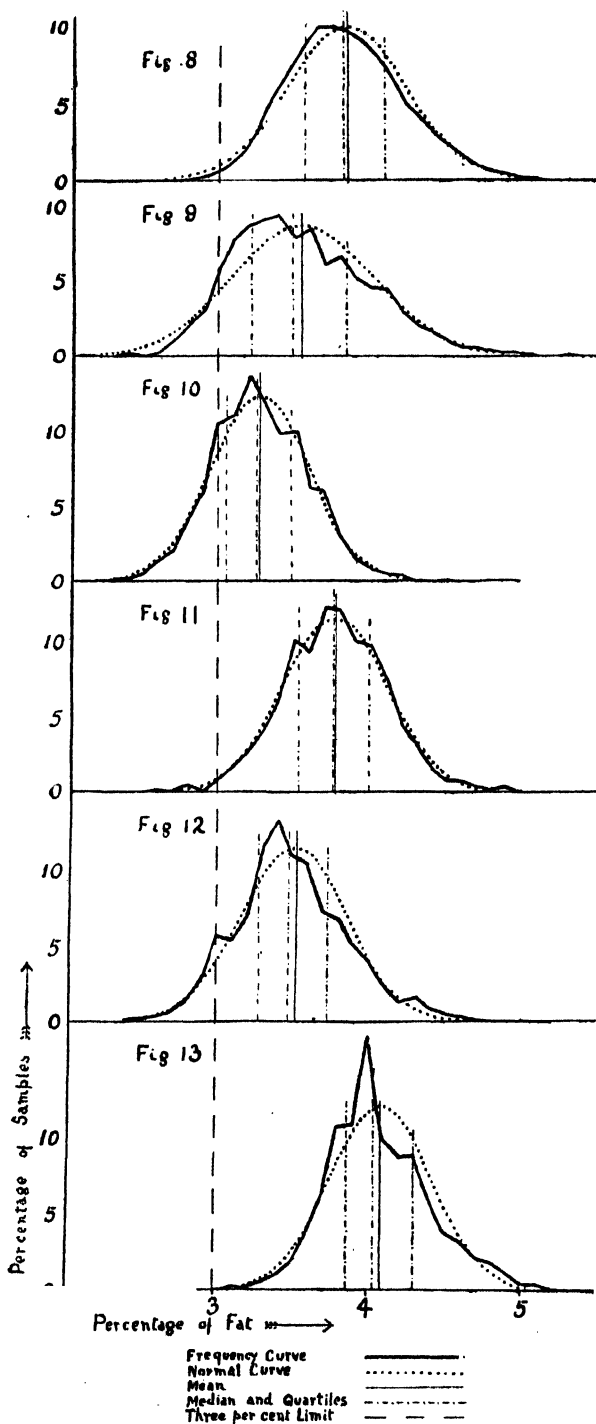


FIG. 4.—Lanark evening milk. 757 samples from single cows. Probable variation, 0.382.

FIG. 5.—1,292 samples from pedigree Shorthorns. Richmond: Committee on Milk and Cream Regulations, 1901, Appendix XXIV. Probable variation, 0.399.

FIG. 6.—2,549 samples from dairy Shorthorns. Richmond: Committee on Milk and Cream Regulations, 1902, Appendix XXIV. Probable variation, 0.493.

FIG. 7.—1,493 samples from Kerry cows. Richmond: Committee on Milk and Cream Regulations, 1901, Appendix XXIV. Probable variation, 0.599.

FIG. 8.—76,058 mixed milk samples as received by Aylesbury Dairy Company from 1894–99. Richmond: Committee on Milk and Cream Regulations, 1901, Appendix XXV. Probable variation, 0.267.

FIG. 9.—Dairy Company "A." 5,600 mixed milk churn samples, morning and evening, over period October 1, 1912, to June 30, 1913. Probable variation, 0.314.

FIG. 10.—Dairy Company "C." 1,499 samples of mixed morning milk from churns. One year period. Probable variation, 0.216.

FIG. 11.—Dairy Company "C." 1,417 samples of mixed evening milk from churns. One year period. Probable variation, 0.231.

FIG. 12.—Dairy Company "B." 4,377 samples of mixed morning milk from churns. One year period. Probable variation, 0.233.

FIG. 13.—Dairy Company "B." 2,460 samples of mixed evening milk from churns. One year period. Probable variation, 0.222.

It will be seen that where the milk of single cows is concerned, the correspondence with the normal curve is quite marked. Naturally, the greater the number of samples, the smoother the curve. The results obtained at Lanark, and published by Dr. Wilson, Medical Officer of Health for Lanark in 1913, comprised 1,511 samples equally divided between morning and evening. The total number of cows was 118, comprised in four separate herds at different farms. The samples were taken over short periods of three to four days each during November and December, and in one case fortnightly sampling was carried out as well. Great pains were taken to make the milking intervals as even as possible. The conditions were therefore very different to those obtaining in the case of the Moundsmere herd results, and there was plenty of opportunity for the intervention of factors which might make the variation abnormal. Allowing for the difference in the means, the distribution is seen to be remarkably similar to that of the Moundsmere results. The other frequency curves for the milk of single cows are drawn from Richmond's results published in the Report of the Milk and Cream Regulations Committee, 1901.

Mr. Richmond informs me that these results were obtained from the Aylesbury Dairy Company herds at Horsham, samples being taken at intervals extending over many months. The analyses were carried out by Dr. Vieth. Both morning and evening milk of the same day are included in these series, which is probably the reason why the relative dispersion is so high in two of them. The greater the difference between morning and evening milk the higher, of course, will be the dispersion if all the results are taken together.

It is noticeable that in some of these curves for single cow milks a certain degree of skewness is evident—that is to say, the point of maximum density of the results falls more or less to the left of the mean. This may be due to some extent to the way in which the curves have been fitted to each other, but is more probably an example of the slight asymmetry which is found to characterise many series of physiological variations.

No general rule can be formulated from so few instances, but the similarity of the Moundsmere and Lanark curves leads one to suspect that if sufficient samples are taken from corresponding milkings of a fair number of cows at one farm over a short period, the fat percentages will be found to follow approximately the law of normal variation, with a probable error of about 0.38 or 0.39.

When we come to the consideration of the frequency curves for mixed milk samples, it is evident that in several cases a greater degree of skewness is met with (*cf.* Richmond, *ANALYST*, 1904, 29, 180, and Arup, Huish and Richmond, *ibid.*, 1917, 42, 118). The records were kindly placed at my disposal by several large dairy companies, and, as far as I have been able to ascertain, comprise only genuine milks as received from the farms in churns. The normal curve for the probable variation in each case is shown by the dotted line. The left-hand branch of the curve is, in most cases, appreciably steeper than the right-hand branch. This is especially pronounced in Fig. 9.

This asymmetry may be partly explained by reference to the figures for fat percentage and milk yield in Table I. If we have a number of cows in varying stages of the lactation period, such as will ordinarily be found in a herd, the milk fat varies

very roughly in inverse proportion to the yield ; but, as lactation progresses, the yields fall off much more rapidly than the milk fat increases. If therefore the samples are mixed at random, as would be done commercially in the filling of churns, the samples low in fat will, by reason of their relatively large bulk, exercise a preponderating influence upon the final series of mixed milk samples, and the result will be that an undue proportion of samples will fall just below the mean. In the case of the evening milks this relation between milk fat and yield is not so pronounced, so that there should not be the same tendency for the frequency curve of the mixed milk samples to assume an unsymmetrical form. This may be a partial explanation of the differences observed by Richmond between the curves for morning and evening milk, in addition, possibly, to the one suggested by him of a fortuitous admixture of morning and evening churns in one consignment. The relationship between morning and evening curves seems, however, to be somewhat erratic. Thus in Figs. 12 and 13 the asymmetry is, if anything, greater in the evening than in the morning milk, while in Fig. 9, where morning and evening samples are taken together, it is very pronounced.

An actual numerical expression of the degree of skewness or asymmetry is given by the expression

$$\frac{\text{Mean—middle point}}{\text{Probable variation.}}$$

The values of this expression for all the series of milk records dealt with in this paper are as follows, arranged in descending order :

Mixed Milk.	Single Cow Milks.	Degree of Skewness.
Dairy Company—"B" evening ...		0.234
Dairy Company—"B" morning ...		0.167
Dairy Company—"A" all samples		0.159
	[Richmond—dairy Shorthorns ...	0.146]
	[Richmond—Kerrys ...	0.128]
	Lanark—evening milk ...	0.105
Richmond: Milk Committee, 1901		0.094
Dairy Company—"C" morning ...		0.092
	Moundsmere—evening milks ...	0.081
	Lanark—morning milks ...	0.079
	Richmond—pedigree Shorthorns	0.043
	Moundsmere—morning milks ...	0.026
Dairy Company—"C" evening ...		0.022

Although there are several irregularities here, it is evident that there is a definite tendency for mixed milk samples to show a greater degree of skewness in the frequency curve than is the case with single cow samples. This tendency becomes still more marked if we neglect the Kerry and dairy Shorthorn series given by Richmond, which show what may perhaps be regarded as an abnormally high

degree of dispersion. It is noticeable that the asymmetry lies in every case in the same direction—*i.e.*, the middle point is always lower than the mean.

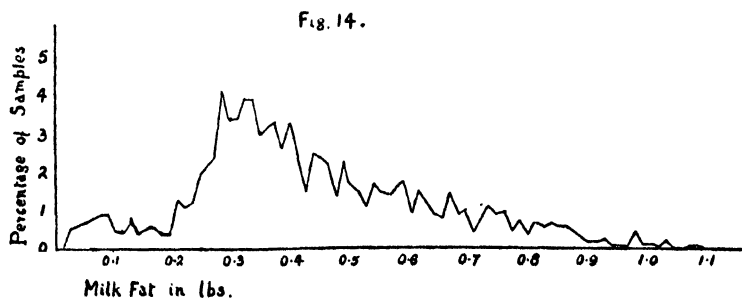
The small number of the series dealt with above, and the fact that the single cow series refer for the most part to short periods and individual farms, while the mixed milk series cover the whole year and are drawn from many farms, render the comparison uncertain. To establish definitely the extent to which varying yield affects the distribution of fat in series of mixed milk samples would require further series of records taken under more comparable conditions.

The asymmetry of these curves is of interest from the point of view of the present presumptive limit for milk fat. Thus, the Aylesbury Dairy records for 1900 to 1912 show that the average fat content for May and June was 3·31 per cent. and 3·28 per cent. respectively. At the same time the average number of samples falling below 3 per cent. was quite small, as shown in Table VII., where the figures show the actual percentage of samples falling at different fat percentages in May and June. If it were not for the skewness of the frequency curve, and the consequent steepness of the left-hand branch, the number of samples falling below 3 per cent. would be much greater.

TABLE VII.—AYLESBURY DAIRY AVERAGES, 1900-1912.

	Below 2·7 per Cent. Fat.	2·7 to 2·8 per Cent. Fat.	2·8 to 2·9 per Cent. Fat.	2·9 to 3 per Cent. Fat.	Total below 3 per Cent. Fat.
May	0·24	0·68	1·76	3·33	6·01
June	0·19	0·54	1·48	3·78	5·98

It is, perhaps, of interest to note that the figures in Table VII. increase very roughly in geometrical progression. Assuming this rate of increase to hold good approximately both for Aylesbury Dairy milk and for the mixed morning milk of the Moundsmere herd, the probability is that the latter, with the mean standing



at 3·08 per cent., if mixed in churns in the ordinary way, would have shown something like 25 per cent. of mixed milk samples below 3 per cent. of fat. This, of course, is only a very rough estimate.

In Fig. 14 is shown the frequency curve of the actual yield of milk-fat in the

morning's milk of individual cows of the Moundsmere herd for the period April 18 to May 26, 1915. This shows to a remarkable degree the combined effect of quality and quantity. It is seen that the weight of milk-fat yielded by a cow at one milking is very seldom less than 0.25 pound, but that the limit towards the upper end of the scale is not nearly so sharp. The "mode," or point at which the greatest density of results occurs, lies far to the left of the mean.

DISCUSSION.

The PRESIDENT (Mr. Smetham) said that in 1891 and 1892 he carried out a series of investigations into Cheshire cheese-making at the Worleston Dairy Institute under the auspices of the Board of Agriculture. The herd, which consisted of thirty Shorthorns, was arranged to calve about the same time. In the course of the investigation not only were average samples taken of the night and morning milk, but also a considerable number of single samples were also taken and analysed, and observations of temperature and other meteorological conditions were duly registered, to see to what extent alterations in conditions affected the milk, and, incidentally, the quality and quantity of cheese. The milk gradually decreased from the time the experiment was started till the middle of October, when the experiment ceased. Generally speaking, the flow of milk fell off towards the end of the period, but the quality of the milk, particularly with regard to fat content, improved very considerably. When a period of cold weather set in, the quality of the milk was very seriously affected, and similarly the quality and quantity of the cheese. From his own experience he found that his results were in the main in accordance with the results given by Dr. Monier-Williams. Referring to the series of figures given in Richmond's records, he said that the gradual elimination of the poorest supplies would probably affect to some extent the averages.

Captain J. GOLDING was of opinion that the title of the paper might lead to some misunderstanding in dairying circles, where the term "milk records" was reserved for a catalogue of the performance of individual cows as to quantity of milk yielded, and did not always include the fat content. He did not think the percentages of fat in the milk of recently calved cows (Group 1) were remarkably low, having in view the times of milking. It was by no means unusual to find similar results on testing the milk of individual cows milked at similar intervals. Uniformity in the composition of this important foodstuff was, he thought, unattainable without undesirable artificial sophistication. The natural product from different breeds and from herds milked at different times could not be uniform in composition, and, like other goods of varying quality, could only be controlled by a guarantee given by the seller, who should be able to obtain a varying price according to quality.

Mr. HAWKINS pointed out that it was sometimes alleged by the defence in cases of deficiency of fat that the turning out to grass was the main cause of such deficiency, but the figures recorded by Dr. Monier-Williams did not appear to support any such contention.

Mr. H. DROOP RICHMOND wrote: By the courtesy of Dr. Monier-Williams I have been enabled to study his paper on Milk Records, and as a previous worker on the subject have found it of great interest and value. Mention is made of some

results which, though attributed to me, are really the work of Vieth, the analysis of all the milk of single cows having been carried out by him, and it is only the circumstance that I presented the records to the Milk Standards Committee as representative of the Aylesbury Dairy Company that has led to their being associated with my name. All the single cows belonged to the herds of the Aylesbury Dairy Company at Horsham, and samples were taken at intervals extending over many months, both morning and evening milk of the same day being analysed. The analyses were made by determining total solids and sp. gr., and calculating the fat by the formula devised by myself, Dr. Vieth being responsible for the analysis and I for the calculations.

I am not quite in agreement with Dr. Monier-Williams in his deductions from the Aylesbury Dairy figures given in Table VII., which are means of thirteen years' observation, and are derived from a total of nearly 10,000 analyses in each month, a number probably sufficient to give fairly accurate data. He says: "if it were not for the skewness of the frequency curve and the consequent steepness of the left-hand branch, the number of the samples falling below 3.0 per cent. would be much greater."

I have calculated by the theory of probabilities what the figures should be, and found that the observed and calculated figures are in remarkably close agreement, being—

	May.		June.	
	Observed. Per Cent.	Calculated. Per Cent.	Observed. Per Cent.	Calculated. Per Cent.
2.9-3.0	3.33	3.40	3.78	3.53
2.8-2.9	1.76	1.48	1.48	1.48
2.7-2.8	0.68	0.72	0.54	0.54
Below 2.7	0.24	0.28	0.19	0.19

The most interesting point brought out in the paper is the skewness of the curves, a point which is not fully elucidated; it has been overlooked that skewness of the curves necessarily follows, firstly, if the results can be arranged in a limited number of series, having different means and probable variations, and tends to disappear as the number of series multiplies; secondly, if the value of the interval taken varies progressively; and thirdly, if there is a disturbing cause which does not affect different results to the same degree—a condition which is not very different from the second.

All these conditions operate in milk records; I have shown (ANALYST, 1904, 29, 180) that the skewness of the curve for six years' records (*cf.* table on p. 212, where it is referred to as Richmond: Milk Committee, 1901) is practically removed by splitting it up into two equal series representing morning and evening milks respectively, and later, in conjunction with Arup and Huish (*ibid.*, 1917, 42, 118), that the skewness of the morning milk curve for 1914 can be eliminated by splitting it up into two series, one greater than the other, the evening curve showing no appreciable skewness.

The second condition is due to the Gerber results being slightly low with high fats, owing to the fact that an 11 c.c. pipette delivers less of a rich milk than a poor milk (ANALYST, 1905, 30, 326; *cf.*, Day and Grimes, *ibid.*, 1918, 43, 124); the difference is not enough to affect individual determinations, but, as the effect is cumulative, it will have a slight effect in skewing the curve on the right-hand branch.

The third condition appears in sampling errors owing to cream rising, and may best be illustrated by mentioning that, while the minimum amount of fat in milk cannot be as low as 0, the maximum may be very much more removed from the mean; if sampling errors were absolutely fortuitous the high results would be higher in proportion than the low. It is probable, however, that more samples are taken from near the surface of milk than from near the bottom, and if the milk is not uniform the proportion of high samples will tend to be greater than that of the low. This fact, while not affecting appreciably the mean value, will tend to skew the curve appreciably on the low side.

I do not think that the fat percentage and milk yield will have any appreciable effect on the asymmetry if a sufficiently large number of results be taken, though it will undoubtedly in a small series; the whole theory of probabilities, and the calculation of probable error and dispersion of results, is based on the assumption that the variations are affected by fortuitous circumstances, and that there is no constant circumstance affecting some results more than others; in a large series milk yields and fat percentages will tend to vary independently and not go together.

Asymmetry is due to the conditions affecting different series of results to a different extent, such as the conditions mentioned above, as well as also variation due to breed, feeding, times of milking, etc., on different farms, and to the number of series differently affected not being sufficient for the conditions affecting them to approximate to fortuity.

I would mention that variations in conditions affect the mean $\left(\frac{\Sigma(v)}{n}\right)$ least, the probable variation $0.6745 \sqrt{\frac{\Sigma(v^2)}{n-1}}$ next, and the distribution $\frac{1}{\sqrt{\pi}} \left(\int_0^x e^{-\frac{t^2}{2}} dt \right)$ to the greatest extent; while the mean of a series is probably of a high order of accuracy, and the probable deviation a fairly exact figure, the distribution on which the asymmetry depends is too sensitive to conditions, which can often only be guessed at, to be of much importance.

Both Vieth and I have studied carefully the effect on the mean of our method of deducing our average percentage by averaging the morning and evening means; the proportion of morning to evening milk over a long series of years was found to be 100 to 85, and we have found that the difference between the true mean and the average of the means of the two meals is less than 0.01 per cent. We concluded that the varying quantities represented by each sample do not affect the mean of the fat in the samples to any appreciable extent. It is not correct to take the yield of the Aylesbury Dairy Company morning milk as 50 per cent. greater than that of the evening milk; the mean milking intervals were 13.2 and 10.8 hours, and in both Aylesbury Dairy Company and Moundsmere cases the yields and intervals corresponded approximately.

Dr. Monier-Williams, in reply, agreed that one of the main reasons for the occurrence of poor milk was unevenness in milking intervals. Milking hours depended upon labour, transport, and market requirements, and uneven intervals seemed to be in many cases unavoidable under the existing system. There was a great difference observable in the records from different farms supplying the same dairy company; some farmers appeared to be able to keep the supply up to a uniformly high level, while others in the same district were not so successful. Records of fat percentage in milk from the north and the south of England, taken over corresponding periods in the same year, were practically identical, which seemed to show that, in the long run, differences in climate had little if any effect on the fat content.

With regard to Captain Golding's remarks as to the low fat content of milk from newly calved cows, the figures given in Group 1 were the averages for twenty-four cows which had calved at periods varying from three-and-a-half to fourteen weeks before the commencement of the experiments. They could not therefore be taken as referring only to newly calved cows.

There was certainly a fall in fat percentage when the cows were turned out to grass, but this was comparatively slight. It was generally accepted that variation in feeding, within ordinary limits, had very little effect on the quality of milk. The daily records of the Moundsmere herd did not show any sudden drop in fat content when the herd was put out to grass.

The points raised by Mr. Richmond called for careful consideration. He remarked that skewness necessarily resulted if the results could be arranged in a limited number of series having different means and probable variations, and tended to disappear as the number of series multiplied. But any series of results, however asymmetric, might be conceived as being divisible into several different "sub-series," each of which might show normal variation, but with the means and probable variations differing according as the original curve showed a greater or less degree of skewness. The division of a skew series in this way into two or more sub-series did not necessarily throw any light on the cause of the original skewness, unless some particular significance could be assigned to the sub-series—*e.g.*, unless they could be proved to represent morning or evening milk. He did not think one was justified in assuming that the sub-series were representative of morning or evening milk without actual experimental evidence to that effect.

The fact that a pipette delivered less of a rich milk than of a poor milk was, no doubt, a contributory cause of the skewness of the curves, but should affect all series to the same relative extent—that is, of course, where the results had been obtained by the Gerber method. He doubted whether it explained the tendency of mixed milk curves to be more asymmetric than those of single cow milks.

Sampling errors again would undoubtedly have some effect in skewing the curve, but this effect would lie in the opposite direction to that arising from pipette error, and would also operate to an equal extent in both mixed and single cow samples.

Mr. Richmond had maintained that, in a large series, milk yields and fat percentages would tend to vary independently and not go together. It was in accord with general experience that, as the cow's lactation period progressed, the milk fell off

in quantity, but became richer in fat. If this was true for a small series of results, it should be equally true for a large series, as it was a condition which, while it might not affect every cow, would affect a fairly constant proportion of cows however great the number taken. He agreed with Mr. Richmond that asymmetry was due to a variety of conditions affecting different series of results to a different extent, but was of opinion that the relationship between yield and fat content was in most cases a controlling factor in the skewness of mixed milk curves, as distinct from those derived from single cow samples.

The figures given by Mr. Richmond as showing the close agreement between observed and calculated results for the proportion of samples falling below 3 per cent. of fat were presumably calculated by means of the formula given by him (ANALYST, 1904, 29, 180). This formula was calculated empirically for a skew curve. It was clear from an inspection of the curves in Fig. 8 that if the actual frequency curve was compared with the normal curve, the statement was substantially correct that it was the skewness of the former which accounted for so few samples falling below the limit.

The difference between the true mean and the average of the morning and evening means would, of course, depend both on the difference in fat content and the difference in yield. In the Moundsmere results the true mean was 3.48, and the average of the morning and evening means 3.59, a difference of 0.11.

In seeking to ascertain the relative importance of the different conditions affecting the composition of milk, it was clearly preferable to study the distribution, which was the most sensitive to these conditions, rather than the mean, which was the characteristic least affected.

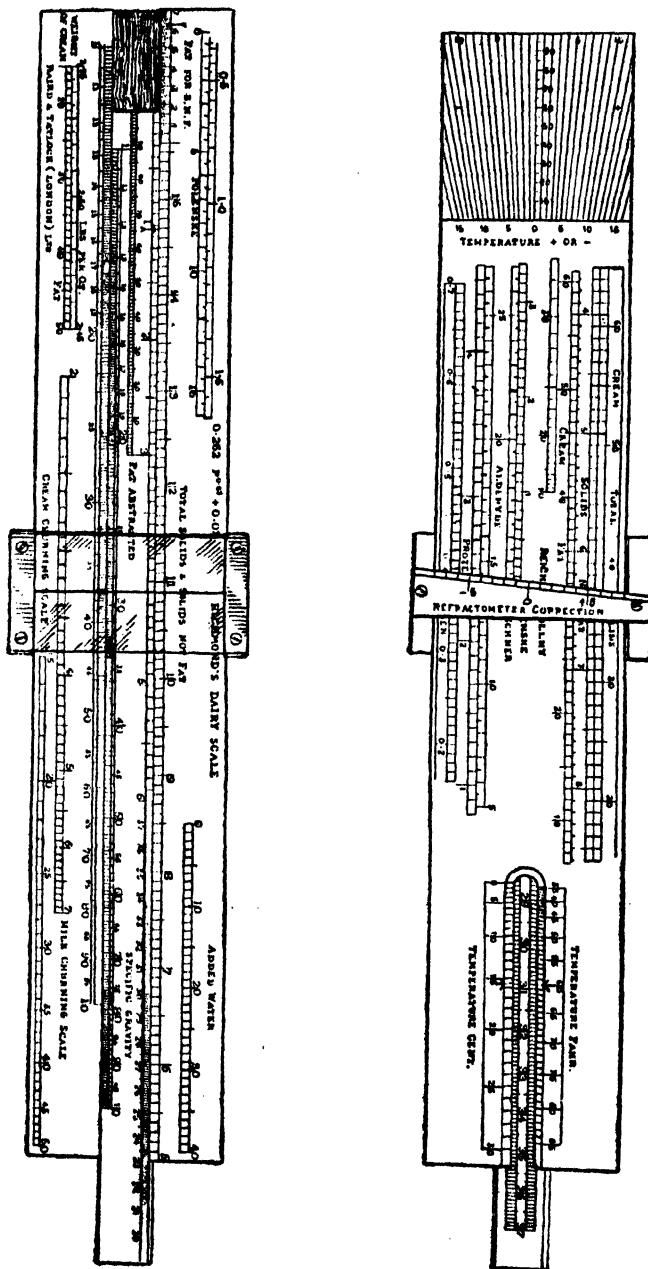


AN IMPROVED SLIDE RULE FOR DAIRY CALCULATIONS.

By H. DROOP RICHMOND, F.I.C.

(*Read at the Meeting, April 14, 1920.*)

In previous papers (ANALYST, 1888, 13, 65; 1894, 19, 38; 1898, 23, 2) I have described the milk scale with various additions, and from time to time I have made other modifications to my own rule. As there are many calculations used in the analysis of milk and milk products, which may be combined in the same rule as the fat-total solid calculation for which the milk scale was designed, I have redrafted the scales on the rule, so that practically every calculation required in dairy analysis can be made with it. On the front of the rule, besides the original calculation of fat from total solids and sp. gr. of the milk scale, solids-not-fat can be deduced from fat and sp. gr., and "fat abstracted" and "added water" scales are also included; the latter reads not only from the solids-not-fat, but also (with an error of 1 in 69) from $G + F$ (*cf. ibid.*, 1898, 23, 69). For ease of reading, a cursor is included,



the celluloid form described by Stokes (ANALYST, 1894, 19, 39) having been adopted. A scale connecting Polenske figures with $0.262P^{0.66} + 0.09$ (cf. Bolton, Richmond and Revis, *ibid.*, 1912, 37, 189) appears in the top left-hand corner.

A 10-inch logarithmic scale occupies the lower portion of the slide; in my experience this simple scale, without the scale of squares on the ordinary slide rule, suffices for practically all analytical calculations. Milk and cream churning scales, for calculating the amount of butter which can be produced from a given number of gallons of milk or quarts of cream of any percentage of fat, lie below the logarithmic scale and are used in conjunction with it. By means of the "weight of cream" scale adjacent, and by introducing the density of the milk, butter calculations can be made from pounds of cream or milk.

On the back of the scale at one end corrections of sp. gr. for temperature can be made, the temperature being given in both Fahrenheit and Centigrade degrees.

At the other end, butyro-refractometer readings can be corrected to a standard temperature by means of the fan-like scale and special cursor; this is modified from the chart previously published (ANALYST, 1907, 32, 46). In the middle of the back, the mutual relations between total solids, fat, and solids-not-fat, in cream are expressed; lower down, the ratios between Reichert-Wollny and Polenske figures and Kirschner and Polenske figures (*ibid.*, 1919, 44, 166) are given, and, at the bottom, aldehyde figures (*ibid.*, 1911, 36, 9) may be converted into proteins and percentages of nitrogen and *vice versa*. The cursor may be reversed to aid the calculations on the back of the scale.

In all, it has been found possible to include some twenty scales, without making the rule unduly large, and, as one of them is a logarithmic scale, practically any calculation can be performed with ease and with an accuracy of three significant figures.



THE EXAMINATION OF CHINESE CRUDE CAMPHOR.

By E. R. DOVEY, A.R.C.Sc., A.I.C.

(*Read at the Meeting, May 5, 1920.*)

In the examination of crude camphor, the estimations usually required are those of non-volatile matter (or dirt), moisture, and oil, and the sum of these impurities subtracted from 100 per cent. is supposed to represent the camphor present. The moisture may be conveniently estimated by the calcium carbide method, allowing three hours for the evolution of gas, and the dirt by the residue left after volatilising a weighed portion of camphor.

So far as is known to the writer, no reliable method for the estimation of camphor oil in crude camphor has been published, other than the melting-point method given in Allen's "Commercial Organic Analysis," vol. iv., p. 197.

As this laboratory was called upon to examine a considerable number of Chinese crude camphors, the following method worked out here may be of interest: The moisture is first estimated on the well-mixed sample, then 100 grms., weighed to the nearest 0.1 gm., are transferred to a press and pressed between two layers of lint. The

press designed for this work has a steel cylinder 2 inches in diameter, and 6 inches deep, and is furnished with a movable perforated bottom plate. The piston is operated by a strong screw thread. The sample is allowed to remain in the press under pressure for fifteen minutes, at the end of which time it is carefully removed and the pressed cake weighed, any camphor adhering to the lint being carefully brushed off and added to the cake. From the loss in weight the amount of water plus oil expressed is found.

The moisture is then estimated on the pressed cake, and from the difference between the result and the original moisture the amount of water expressed is found, and, by difference, the amount of oil in the expressed liquid is found.

It is then assumed that the water still remaining in the pressed cake is associated with as much oil as that in the expressed liquid, and the total oil calculated on that basis. The accuracy of this assumption may be open to question, but, with a good press, very little moisture remains in the cake, while the m. pt. of the pressed camphor usually indicates a fairly high degree of purity.

GOVERNMENT LABORATORY,
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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Acidimetry of Red Wines and Fruit Juices. A. M. Macmillan and A. Tingle. (*J. Ind. and Eng. Chem.*, 1920, 12, 274-276.)—The use of litmus-paper for the titration of acidity in red wines is not very satisfactory, and the results are not comparable with those obtained with white wines when using phenolphthalein. The authors propose to use phenolphthalein also in the titration of red wines, and to determine the end point by spectroscopic means. Twenty-five c.c. of the wine or juice to be examined are introduced into the titration vessel with about 75 c.c. of water. $\frac{N}{10}$ sodium hydroxide solution is then rapidly added from a burette until the end point is near. This may be known either by the change in the natural colouring matter or by a previous rough titration. Phenolphthalein (2 c.c. of a 1 per cent. solution) is next added and the titration is continued with ordinary caution, the liquid being examined before a small spectroscope after each addition of alkali. The end point is shown by the sudden appearance of the absorption band characteristic of phenolphthalein. It lies in the green region of the spectrum very close to the yellow. The thickness of liquid suitable for observations at the dilution specified is 30 to 35 mm.; the liquid may be examined in flat 6-ounce bottles such as are used for retailing small quantities of whisky. The dilution of the sample before titration may be varied according to the depth of colour and the volume of standard alkali likely to be required in the titration. Sufficient water must be added to a highly coloured liquid to reduce the effective concentration of its colouring matter and sufficient indicator must be added to give an effective concentration showing the characteristic change. The region of the spectrum in which the absorption band due to the change of colour of the indicator is situated must not be obscured by absorption bands due to the original colouring matter of the liquid. J. F. B.

Boric Acid Modification of the Kjeldahl Method for the Analysis of Cereals and Soils. F. M. Scales and A. P. Harrison. (*J. Ind. and Eng. Chem.*, 1920, 12, 350-352.)—Winkler's method of using boric acid instead of sulphuric acid for the fixation of the ammonia distilled in Kjeldahl's method (*Zeitsch. angew. Chem.*, 1913, 23, 231) gives as accurate results as the older method, and has the advantage that the boric acid solution need only be measured approximately, only one standard solution (for titration) being required. Bromo-phenol blue is a more satisfactory indicator than either methyl orange or Congo red, which were used by Winkler. C. A. M.

Note on the Reichert-Meissl-Polenske Method. H. D. Richmond and G. F. Hall. (*J. Soc. Chem. Ind.*, 1920, 39, 80r.)—The directions given by Polenske (*ANALYST*, 1904, 29, 154) are frequently modified by varying the time required for the distillation, and cooling the distillate in water at 15° C. instead of the prescribed 10° C. Experimental determinations upon samples of coconut oil and butter fat

have shown that the time of distillation (from nineteen to thirty-eight minutes) does not make any material difference in the case of butter fat, but that with coconut oil it is essential to adhere to Polenske's directions (twenty-two minutes). On the other hand, the temperature to which the distillate is cooled (5° to 20° C.) does not have much influence on the result.

C. A. M.

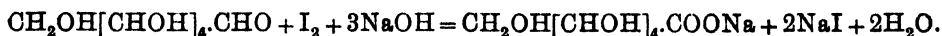
Double Polarisation Methods for the Determination of Cane Sugar. G. W. Rolfe and L. F. Hoyt. (*J. Ind. and Eng. Chem.*, 1920, 12, 250-253.)—In the estimation of cane sugar by the Clerget method an error is caused by the difference in polarisation values in neutral solution for the direct polarisation, and in strongly acid solution for the invert reading. It is desirable, therefore, to make both readings in the presence of the same degree of acidity, but, if hydrochloric acid be employed, its inverting action in the cold is so rapid that accurate results cannot be obtained for the direct polarisation. The authors have investigated both trichloro- and monochloro-acetic acids and find that the latter acid may be employed as the basis of a satisfactory method. The normal weight of sample is dissolved in a 100 c.c. flask, the solution is clarified with an appropriate quantity of lead acetate, made up to 100 c.c. and filtered. Fifty c.c. of the filtrate are transferred to a 100 c.c. flask, 15 c.c. of a 20 per cent. solution of monochloro-acetic acid are added, and the volume is made up to 100 c.c. Direct polarisation must be made within fifteen minutes after adding the acid. For inversion, 50 c.c. of the liquid are placed in a 50 c.c. flask, the cork is tightly tied down and the flask is immersed in boiling water, maintaining active ebullition for thirty minutes, or for sixty minutes in the case of low-grade products clarified with a large amount of lead acetate. The flask is removed, cooled quickly to the temperature of the room, allowed to stand for at least two hours and polarised in the 200 mm. tube with thermometer. All solutions should be made up and polarised as nearly as possible at 20° C. The formula for cane sugar is $S = \frac{2(a-b)}{141 - \frac{t}{2}} \times 100$.

By this method both polarisations are made at the same acidity and sugar concentration. Excess of basic lead acetate equivalent to 0.5 c.c. in a half normal solution of sugar does not affect the inversion or produce troublesome precipitates. The results are more accurate than by the Herzfeld method, and approach more closely to those obtained by the standard invertase method. Inverted solutions of low grade products are lighter in colour than those inverted by the Herzfeld method and therefore easier to polarise, and the error introduced by the necessity of making up to volume after inversion is avoided.

J. F. B.

Iodimetric Estimation of Sugars. H. M. Judd. (*Biochem. J.*, 1920, 14, 255-262.)—When estimating the nature and amount of the carbohydrates found in apples during the process of ripening, in ordinary and cold storage, many difficulties are encountered when using the polarimeter. Since the optical activity of apple juice is very low, and the error involved in calculating the proportion of each of the sugars present from such small readings as 1° and less is very large, an investigation has been made into the quantitative estimation of sugars by means of iodine in

alkaline solution. In the main the results agree with those found by Willstätter and Schudel (ANALYST, 1918, 43, 416), who state that there must be sufficient alkali to neutralise the acid formed as the result of the oxidation, and that the reaction goes best with decinormal solutions in the proportions required by the equation



They point out, however, that in the presence of free alkali the sugars undergo the Lobry de Bruyn transformation, also that cane sugar and lævulose are unacted upon. The present author has found, when working with pure sugars, that neither the method of Colin and Liévin (*Bull. Soc. Chim.*, 1918, 47, 403) nor that of Willstätter and Schudel is exact, and that in no case is the sugar oxidised quantitatively to the corresponding monobasic acid; on the other hand, there is always a small but definite attack on the lævulose. The results obtained with invert sugar are misleading, since the low result obtained from the dextrose is masked by the partial oxidation of the lævulose. Moreover, the reaction mixture always smells strongly of iodoform, showing that some secondary change is occurring. The chief source of error is probably the action of dilute alkalis on sugars, as investigated by Lobry de Bruyn and Van Ekenstein, part of the dextrose being converted into mannose, which is also oxidised by iodine, and part into lævulose. The whole of the sugar present would thus not be oxidisable by iodine, leading to a low result for dextrose, while lævulose itself would yield a certain proportion of both dextrose and mannose, thus probably accounting for the apparent partial oxidation of the lævulose. A complex but definite equilibrium is, however, reached, and it is possible to utilise the method for the analysis of mixtures of dextrose and lævulose, since experiments show that a given weight of dextrose always uses a definite and constant weight of iodine, although not the amount theoretically required, and this also holds true for levulose. It is thus possible to calculate from the iodine value and copper-reducing power of a mixture the proportion of each sugar present. The copper reduction method employed was that of Bertrand. For the oxidation by iodine the Willstätter and Schudel method was used as follows: 10 c.c. of an approximately 1 per cent. solution of dextrose is mixed with 20 c.c. $\frac{N}{10}$ iodine and 30 c.c. $\frac{N}{10}$ caustic soda, the mixture being allowed to stand fifteen to twenty minutes at room temperature. It is then acidified with dilute sulphuric acid and titrated with thiosulphate. One grm. of dextrose requires 1.315 grms. of iodine. The same volume of 1 per cent. lævulose solution, when treated with 10 c.c. of $\frac{N}{10}$ iodine and 20 c.c. of $\frac{N}{10}$ caustic soda, shows that 1 grm. of lævulose requires 0.1028 grm. of iodine. These constants, when combined with the cupric reducing power of the sugar, may then be used as follows:

- 2.369 = weight of CuO equivalent to 1 grm. of dextrose;
- 2.369 = weight of CuO equivalent to 1 grm. of lævulose;
- 1.315 = weight of iodine absorbed by 1 grm. of dextrose;
- 0.1028 = weight of iodine absorbed by 1 grm. of lævulose.

Let x = weight of dextrose in a given volume, V ;

Let y = weight of levulose in a given volume, V ;

Then $2.369x + 2.369y$ = weight of CuO corresponding to V ;

$1.315x + 0.1028y$ = weight of iodine corresponding to V .

The solutions used were :

- $\frac{N}{10}$ iodine ; 1 c.c. = 0.01298 I ;
 $\frac{N}{10}$ thiosulphate ; 1 c.c. = 0.0128 I ;
 $\frac{N}{10}$ KMnO_4 ; 1 c.c. = 0.06892 Cu.

Mixtures containing known amounts of dextrose and lævulose were analysed in the above manner, and results obtained of a high order of accuracy. The method has also been tried with other sugars with interesting results. The other hexose sugars behave in a manner similar to, but not identical with, dextrose. Each sugar appears to have its own characteristic iodine value, and mannose is oxidised with greater difficulty. Of the pentoses tested, arabinose takes up almost the theoretical amount of iodine, while rhamnose is oxidised much less completely. This may be associated with the fact that it does not, when oxidised, yield the very unstable rhammonic acid, but a lactone. Remarkable results are obtained with the disaccharides. The action on saccharose is negligible. Maltose behaves quite normally, and the iodine value found by experiment agrees closely with that calculated from the equation



Lactose, on the other hand, takes up twice as much iodine as is required by the equation, suggesting that the sugar is hydrolysed, and that both the dextrose and galactose molecules are then oxidised.

H. F. E. H.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Estimation of Chlorides in Whole Blood. J. H. Austin and D. Van Slyke. (*J. Biol. Chem.*, 1920, **41**, 345-348.)—The method of Van Slyke and Donleavy for the estimation of chlorides in plasma (*J. Biol. Chem.*, 1919, **37**, 551) yields results from 30 to 40 per cent. too high when used with whole blood. The following modification is employed, and gives results in agreement with those obtained by the Carius method: 3 c.c. of blood and 15 c.c. of water are measured into a 60 c.c. flask, 30 c.c. of saturated picric acid solution are added, the whole diluted to 60 c.c., mixed, left to stand ten minutes, and filtered. To 40 c.c. of the filtrate 10 c.c. of a solution containing 5.812 grms. of silver nitrate and 250 c.c. of nitric acid per litre are added, the solutions mixed and allowed to stand overnight. The supernatant liquid is filtered, 20 c.c. are titrated, and the result calculated, as in the Van Slyke-Donleavy method. This modification also yields accurate results with plasma coloured by the hæmolytic of the blood cells, and which gives abnormal results by the original unmodified method.

T. J. W.

Determination of Fibrin, Globulin, and Albumin Nitrogen in Blood Plasma. G. E. Cullen and D. Van Slyke. (*J. Biol. Chem.*, 1920, **41**, 587-597.)—*Fibrin nitrogen*: To 5 c.c. of plasma from oxalated blood are added 150 c.c. of 0.8 per cent. sodium chloride and 5 c.c. of 2.5 per cent. calcium chloride, and allowed to stand fifteen minutes and filtered. The residue is washed five times with 0.8 per

cent. sodium chloride, allowing each portion to remain in contact with the fibrin for ten minutes; then transferred to a Kjeldahl flask, and the nitrogen determined. *Filtrate nitrogen*: 20 c.c. of water and 25 c.c. of saturated ammonium sulphate are added to 5 c.c. of plasma, allowed to stand overnight, and filtered. Twenty c.c. of the filtrate are transferred to a Kjeldahl flask, 300 c.c. of 50 per cent. alcohol, 3 grms. of magnesium oxide, and 1 c.c. of white mineral oil are added, and the whole distilled until the distillate gives a negative test with red litmus-paper. The residue in the flask is digested with sulphuric acid and potassium sulphate to a light brown colour, washed down with a few c.c. of water, 10 c.c. more of sulphuric acid added, and the digestion continued for about three hours. The nitrogen is then estimated as usual. *Non-protein nitrogen*: A 50 c.c. flask is half filled with 2.5 per cent. trichloroacetic acid solution and 5 c.c. of plasma are added, the flask filled to the mark with trichloroacetic acid solution, the contents mixed, allowed to stand one hour, and filtered. The filtrate is measured, transferred to a Kjeldahl flask, and the nitrogen determined as above. *Total plasma nitrogen*: This is determined by the Gunning-Kjeldahl method, using 2 c.c. of plasma and digesting three hours after clearing. Corrections for the reagents used should be made. Albumin nitrogen is calculated by deducting the non-protein nitrogen from the "filtrate nitrogen." Globulin nitrogen is determined by subtracting the sum of the "filtrate nitrogen" and the fibrin nitrogen from the total nitrogen. Descriptions are given of the working out of these methods, and the results obtained are tabulated. T. J. W.

Determination of Sugar in Blood. O. Folin and H. Wu. (*J. Biol. Chem.*, 1920, 41, 367-374.)—A modification of the method previously described by the authors (*J. Biol. Chem.*, 1919, 38, 106), in which the phenol reagent is replaced by one containing molybdic acid and sodium tungstate. Two standard solutions are employed containing respectively 1 and 2 mgrms. of dextrose or invert sugar per 10 c.c. These are prepared by dilution of a 1 per cent. stock solution preserved with xylene or toluene. Two c.c. of the tungstic acid blood filtrate are added to a special form of test-tube, and the same volume of the standard solutions to two similar tubes. To each tube 2 c.c. of an alkaline copper solution (prepared by dissolving 40 grms. of pure anhydrous sodium carbonate in about 400 c.c. of water, adding 7.5 grms. of tartaric acid, 4.5 grms. crystallised copper sulphate, and diluting to 1 litre) are added, and the tubes heated in a boiling water bath for six minutes, followed by rapid cooling in cold water without shaking. Two c.c. of the molybdate-tungstate reagent (35 grms. of molybdic acid and 5 grms. of sodium tungstate in 400 c.c. of 5 per cent. sodium hydroxide: the solution is boiled vigorously for about thirty minutes, cooled, diluted to about 350 c.c., and 125 c.c. of concentrated 85 per cent. phosphoric acid added, finally diluting to 500 c.c.) are run into each tube to dissolve the cuprous oxide; the volume is then made up to 25 c.c. with water, the contents of each tube mixed, and the blue colours compared. A special form of test-tube is described which reduces the oxidation of the precipitated cuprous oxide by the air, and tables are provided showing the effect of various factors upon the results. The new modification yields results slightly lower than those given by the original method.

T. J. W.

Estimation of Magnesium in Blood. W. Denis. (*J. Biol. Chem.*, 1920, **41**, 363-365.)—Five c.c. of citrated plasma, serum, or whole blood are run into 15 c.c. of 6.5 per cent. trichloroacetic acid solution, shaken, allowed to stand at least thirty minutes, and filtered. Ten c.c. of the filtrate are used for the estimation of calcium by the method of Lyman (*J. Biol. Chem.*, 1917, **29**, 169), and the supernatant liquid and washings are transferred to a platinum dish, 3 c.c. of 10 per cent. sulphuric acid added, evaporated to dryness, and ignited until white. The residue is dissolved in about 5 c.c. of water, and 10 per cent. hydrochloric acid added drop by drop until the solution is acid to methyl orange. The solution is transferred to a beaker, evaporated to 2 or 3 c.c., concentrated ammonia is gradually added until the solution is alkaline and 0.5 c.c. of 10 per cent. ammonium phosphate solution containing 50 c.c. of concentrated ammonia per litre, the mixture then being allowed to stand overnight. It is then transferred to a conical centrifuge tube, and the beaker washed with 20 per cent. alcohol containing 50 c.c. of ammonia per litre. After centrifuging, the liquid is removed and the beaker again washed with about 10 c.c. of the alcohol-ammonia mixture, this being repeated three times. After removing the last portion of washing liquid the tube is placed upon a water-bath until the ammonia present has evaporated, and the precipitate is dissolved in 10 c.c. of $\frac{N}{10}$ hydrochloric acid, transferred to a 100 c.c. flask, diluted to that volume with water, and mixed. Twenty-five c.c. of this solution are diluted to 50 c.c., 25 c.c. of strychnine molybdate reagent (see Bloor, *J. Biol. Chem.*, 1911, **32**, 34) are added, and, after standing five minutes, the volume of suspension is compared with that produced by 0.01 mgrm. of magnesium in 50 c.c. to which 26 c.c. of the strychnine molybdate solution is added, and which has been allowed to stand for the same time as the unknown. Estimations made by the above method with aqueous solutions, plasma, and serum containing from 0.02 to 0.1 mgrm. of magnesium, give an average recovery of 94 per cent. Estimations made on human blood serum, including pathological cases, give figures varying from 0.8 to 3.8 mgrm. per 100 c.c., and on normal serum from 1.6 to 3.5 mgrms.

T. J. W.

Separation of Hydrocarbons by the Aid of Bacteria. J. Tausz and M. Peter. (*Zentralbl. f. Bakter. u. Parasitenk.*, 1919, **49**, 497-554; *Chem. Zentralbl.*, 1920, **91**, II., 264.)—Certain bacteria which attack naphthenes but not paraffins furnish a means hitherto wanting of separating these bodies. *B. aliphaticum*, *B. aliphaticum liquefaciens*, and the paraffin bacterium, are described; these were grown from garden mould in inorganic or organic media to which *n*-hexane, cyclohexane, or paraffin oil had been added. Paraffin bacteria are without action on naphthenes, benzenoid hydrocarbons, and some paraffins—for example, *n*-hexane and *n*-octane—but attack higher paraffins such as hexadecane, triacontane, and tetracontane. The other two bacteria are inert towards cyclic hydrocarbons and hexylene, but attack paraffins and *n*-caprylene and hexadecylene. The destruction is complete, even in presence of inert hydrocarbons. The presence of a very small proportion of aliphatic hydrocarbon in natural naphthenes, or of impurity in artificially prepared specimens, is shown by the clouding, due to bacterial growth, of media to which the hydrocarbon has been added. Naphthenes thus treated possess higher

constants. The method can be used for the detection of paraffins in crude oils and their products, and for the isolation of pure naphthenes. None of the bacteria showed the presence of urease; diffusible lipase and proteolytic enzymes were recognised in *B. aliphaticum* and the paraffin bacterium, diastase in the paraffin bacterium and *B. aliphaticum*, and catalase in both species. The following new values were obtained: 1·3-dimethylcyclohexane, b.-pt. 118°-120° C., D 20° C./4° C. 0·771, $[n]_{D 20^{\circ} C.}$ 1·4258; 1·3·4-trimethylcyclohexane, b.-pt. 139°-140° C., D 20° C./4° C. 0·789, $[n]_{D 20^{\circ} C.}$ 1·4330.

O. E. M.

Braunstein's Modification of the Mörner-Sjöqvist Process for the Estimation of Urea. A. H. Todd. (*Biochem. J.*, 1920, 14, 252.)—Braunstein suggested the use of crystalline or syrupy phosphoric acid in place of sulphuric acid at the "Kjeldahl" stage of the Mörner-Sjöqvist process (in order to prevent hippuric acid and creatinine escaping precipitation and appearing in the final result as urea). This assumes that urea is completely incinerated under the Braunstein conditions, viz., heating at 145° C. for four and a half hours. The author finds that urea-estimation is by no means quantitative under the original conditions, and the temperature is the most important factor. Experiment showed that incineration of the Mörner-Sjöqvist filtrate for fourteen hours at 185° C. with 15 grms. of Kahlbaum's phosphoric acid gives an accurate quantitative estimation of the urea and of the urea only. With creatine and hippuric acid *per se*, no trace of incineration with phosphoric acid occurred when working under these conditions. The actual heating is best performed in an oil-bath, while phosphoric acid may most conveniently be weighed out by melting it in a water oven, and pouring it out into a weighed glass evaporating basin.

H. F. E. H.

Titration of Organic Acids in Urine. D. Van Slyke and W. W. Palmer. (*J. Biol. Chem.*, 1920, 41, 567-585.)—One hundred c.c. of urine are mixed with 2 grms. of finely divided calcium hydroxide, allowed to stand with occasional stirring about fifteen minutes and filtered. To 25 c.c. of the filtrate 0·5 c.c. of 1 per cent. phenolphthalein is added, and $\frac{N}{8}$ hydrochloric acid run in until the pink colour just disappears, when 5 c.c. of 0·02 per cent. tropæolin 00 solution are added with continual shaking, followed by $\frac{N}{8}$ hydrochloric acid which is added gradually from a burette until the colour equals that of a standard solution in a similar tube containing 0·6 c.c. of $\frac{N}{8}$ hydrochloric acid, 5 c.c. of tropæolin 00 solution, and water to a total volume of 60 c.c. In calculating the required result, a correction is first made by deducting the value obtained in a blank experiment, and finally by deducting the equivalent in organic acids of the creatinine present in the urine. A 0·1 M solution of creatinine is equivalent to a $\frac{N}{10}$ solution of organic acid. Other indicators, including methyl orange, tetrabromophenolsulphonaphthalein and dimethylamino-azobenzene, may be used in place of tropæolin 00. The average 24-hour excretion of healthy adult males is 6 c.c. of $\frac{N}{10}$ acid per kilo of body weight. The theoretical basis of the method is discussed and numerous tables are provided showing results obtained by the above method with various acids, bases, and salts contained in urine, together with determinations made on normal and pathological urines.

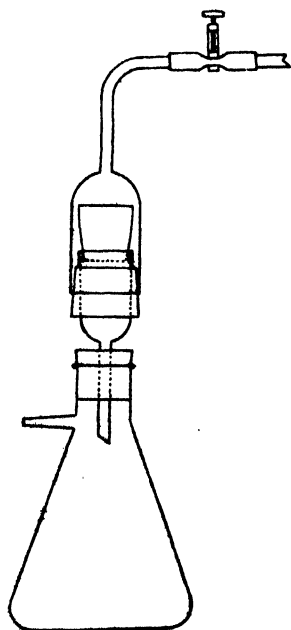
T. J. W.

ORGANIC ANALYSIS.

Estimation of Acetylene in Gaseous Mixtures. J. A. Muller. (*Bull. Soc. Chim.*, 1920, 27, 69-71.)—The following method of estimating acetylene by precipitation as cuprous acetylide gives accurate results. From 70 to 80 c.c. of the gas containing not more than 10 to 11 c.c. of acetylene are shaken in a stoppered flask with an ammoniacal solution of cuprous chloride (1.2 to 1.3 grms. of 50 per cent. cuprous chloride, 6 c.c. of ammonia solution and 5 c.c. of water), until the acetylene is completely precipitated. The flask is then nearly filled with 10 per cent. sodium chloride solution, the liquid acidified with acetic acid, and the acetylide separated and washed. The filter and its contents are treated drop by drop with strong sulphuric acid until a homogeneous mass is obtained, and heated in a crucible (which is nearly covered) until acid fumes cease to be evolved, after which the temperature is raised to redness, so as to convert the whole of the copper into cupric oxide. The weight of the latter multiplied by 0.1407 gives the corresponding volume in c.c. of acetylene.

C. A. M.

Estimation of Cellulose in Woods. W. H. Dore. (*J. Ind. and Eng. Chem.*, 1920, 12, 264-269.)—The estimation of cellulose in wood and similar vegetable tissues by the chlorination method may be made in two ways: with previous hydrolysis of the hemicellulose and furfural-yielding groups or without hydrolysis. In the former



case the hydrolysis may be effected either by boiling with alkali, as in Cross and Bevan's method, or by acids at high temperatures, as in König's and Johnsen and Hovey's methods; chlorination without hydrolysis was originally recommended by Renker (*ANALYST*, 1910, 35, 71), and has been adopted by several later investigators. There is a considerable difference in yield of cellulose estimated in these two ways, and the author has carried out experiments to ascertain whether the lower yield after previous hydrolysis is accounted for simply by the removal of carbohydrates inferior to cellulose, or is due to the destruction of the true cellulose. In these experiments 2 grms. of the wood, finely ground, were extracted, first with benzene and then with alcohol, for six hours each. The chlorination was performed in a Gooch crucible by the Seiber and Walter method in an apparatus illustrated in the accompanying figure, the sulphite treatment being conducted in the same crucible. The chlorine treatments, four in number, were carried out for twenty, fifteen, fifteen, and ten minutes respectively. The yields of cellulose corresponding to various pre-treatments were determined as follows:

Renker's method (no hydrolysis), 48.51 per cent.; original Cross and Bevan's method (previous boiling with 1 per cent. sodium hydroxide for one hour), 45.83; Johnsen and Hovey's method (hydrolysis for

four hours with acetic acid and glycerol at 135° C.), 44.25. The ratio of α -cellulose to total cellulose in each of the products was determined by treatment with 17.5 per cent. sodium hydroxide, and showed 0.75, 0.77, 0.78, respectively. The yields of furfural were 0.50, 0.27, and 0.26 per cent. respectively. From these data it was concluded that the hydrolysis methods give substantially lower yields of cellulose than the direct chlorination method, and that the percentage of resistant α -cellulose is practically the same in all the products. Consequently the hydrolysis is not selective towards the inferior carbohydrates, but attacks the α -cellulose in the same ratio. Hence the Renker process, without special hydrolysis, is to be preferred since it gives the maximum yield of resistant cellulose. The above conclusion was confirmed by similar experiments on purified cotton cellulose, in which it was found that Renker's method gave about 4 per cent. more total cellulose than the hydrolysis methods, and that this cellulose was no less pure than the cellulose residue from the methods giving lower yields. It is considered that hemicelluloses and pentosans are hydrolysed and dissolved in the processes incidental to the direct chlorination method, and that special hydrolysis treatments are not only unnecessary, but detrimental to an accurate estimation of the true cellulose.

J. F. B.

Detection of Ligneous Impurities in Cotton and Cotton Waste for Nitration Purposes. F. L. Barrett. (*J. Soc. Chem. Ind.*, 1920, 39, 81-82r.)—The reaction is based on the strong affinity of lignin for basic dyestuffs, towards which it acts as a mordant similar to tannin. Malachite green is recommended for this purpose, and its fixation by the lignin is promoted by the addition of formaldehyde. After dyeing, the green dyestuff loosely held by the cotton fibres is discharged by a bleaching powder solution, to which the dyestuff fixed on the lignin is relatively resistant. The dye solution is prepared by dissolving 0.1 grm. of malachite green in water, diluting the solution to 500 c.c., adding 50 c.c. of 40 per cent. formaldehyde solution and 1 grm. of sodium bisulphate dissolved in a little water, and making up the whole to 1 litre. The bleaching powder solution is made by shaking 20 grms. of bleaching powder with 1 litre of water, allowing to settle and drawing off the clear liquor. In performing the test 300 c.c. of the dye solution are heated in a beaker in a boiling water bath, and 8 grms. of the cotton are steeped in the hot liquor for ten minutes. At the end of that time 25 c.c. of the bleach solution are added, and the liquid is stirred rapidly. The colour of the bath is immediately discharged, and the cotton is allowed to remain therein for a further five minutes. The liquor is then poured off through a funnel, using the cotton as a filter, and the material is rinsed thoroughly and examined, while wet, for the presence and distribution of green specks. It is important to use pure malachite green, and to keep the baths well stirred during dyeing and bleaching.

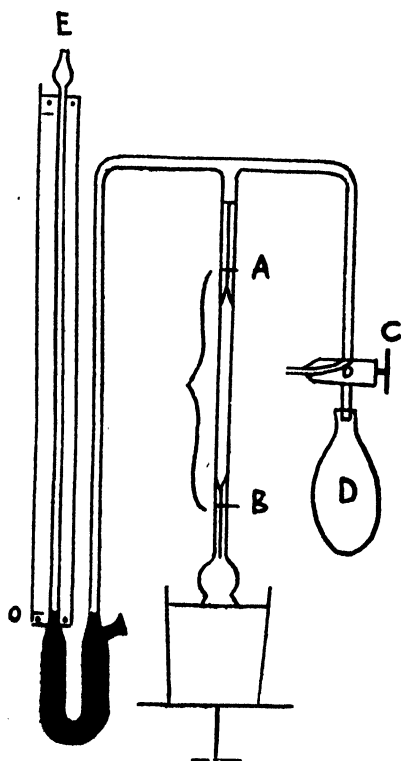
J. F. B.

Distillation Method for the Analysis of Commercial Chlorobenzene. F. Bourion. (*Comptes rend.*, 1920, 170, 933-935.)—Commercial chlorobenzene contains unchlorinated benzene and *p*-dichlorobenzene together with higher chlorinated compounds. The quantities of these impurities present, and that of the actual

monochlorobenzene, may be estimated by fractionally distilling 1,500 grms. of the sample, and collecting the fractions boiling between 80° and 130° C., 130° and 131.5° C., and 131.5° and 172° C. The first and third fractions are then refractionated (at 10° intervals of temperature) until further separation is impossible. The quantities of monochlorobenzene separated from these fractions are added to the main bulk of the monochlorobenzene (b.pt. 130° to 131.5° C.) after a portion of this has been carefully refractionated in order to ascertain the small amount of impurities still retained by it; unavoidable losses during the distillations are distributed proportionally among the various fractions.

W. P. S.

Testing the Strength of Glue Jellies. W. H. Low. (*J. Ind. and Eng. Chem.*, 1920, 12, 355-356.)—A modification of Smith's glue tester is described, in



which some of the errors of the original instrument are obviated. Essentially it consists of a U-tube of the form shown in the diagram. This has a capillary attachment ending in a thistle funnel covered with a rubber membrane, and the pressure is applied by a rubber bulb, D. The U-tube is charged with mercury above which is a layer of coloured water, and an adjustable scale to indicate the pressure is fixed on the limb E. Sufficient water is introduced into the thistle funnel and capillary tube so that when a flat glass plate is held against the mouth of the former, the level of the water will be at the upper mark A, the three-way cock C being open to the air. This cock is then closed to the air, and opened to the rubber bulb and scale tube E, the water in which has been set at the zero mark. Steady pressure is applied to the bulb until the water in the capillary falls from A to B, and the level of the water in the scale tube is then read. In testing a glue jelly the vessel containing it is fixed so that the surface of the jelly is in contact with the rubber diaphragm, the pressure applied, and the readings taken as before. The

difference between the pressure recorded and that previously required to overcome the resistance of the rubber diaphragm alone affords a measure of the consistency of the glue jelly.

C. A. M.

Technical Methyl Chloroformates and their Analysis. M. Delépine. (*Bull. Soc. Chim.*, 1920, 27, 39-45.)—For the estimation of formaldehyde and formic acid in technical methyl chloroformates it is best to determine the former by means of iodine, and then the formaldehyde and formic acid together by means of perman-

ganate, and to obtain the formic acid by difference. A weighed quantity (about 0.4 c.c.) of the sample is shaken in a closed vessel with 50 c.c. of $\frac{N}{1}$ sodium hydroxide solution, and, after 30 minutes, the liquid is made up to 125 c.c. The formaldehyde is estimated by adding 20 c.c. of $\frac{N}{10}$ iodine solution, drop by drop, to this solution with continual shaking, and then, after twenty minutes, adding 1 c.c. of dilute (1 : 5) sulphuric acid, and titrating the excess of iodine with standard thiosulphate solution. The formic acid is estimated by neutralising 25 c.c. of the solution with $\frac{N}{1}$ sulphuric acid (turmeric paper as indicator), then adding 5 c.c. of 10 per cent. sodium carbonate solution, and 50 c.c. of potassium permanganate solution (3.16 grms. per litre). After thirty minutes a measured quantity of oxalic acid solution (6.3 grms. per litre) is added (usually 40 c.c. is sufficient), and then 20 c.c. of the dilute sulphuric acid, and the clear solution titrated with the permanganate solution. In the case of each estimation, blank determinations are made with the same reagents in the absence of methyl chloroformate. The same solution of the sample is used for the estimation of the chlorine and carbon dioxide by the usual methods. Carbon monoxide is estimated by treating 0.3 to 0.5 gm. of the methyl chloroformate with 10 c.c. of 4N-potassium hydroxide solution in a mercury ureometer, and measuring the liberated gas. Methyl formate and chlorocarbonate do not evolve either carbon monoxide or formaldehyde. The results vary with the proportion of chlorine in the sample. Thus, German products containing 58.7 to 58.9 per cent. of chlorine evolved 80 and 73 c.c. of carbon monoxide respectively, whilst French products with 65.8 per cent. of chlorine yielded only 100 c.c. instead of 147 c.c. This is due to the fact that the chlorine in the course of the reactions forms further substitution products.

C. A. M.

Determination of the Tensile Strength of Glue. G. Hopp. (*J. Ind. and Eng. Chem.*, 1920, 12, 856-858).—Solutions of glue of 60 to 80 per cent. by volume are made by soaking the glue for twelve hours, and then melting it up to a temperature of 160° F., care being taken to avoid frothing and air bubbles. The solutions are poured into polished iron moulds, 12 inches square and $\frac{1}{4}$ inch deep, and left to jell for about five hours. The resulting sheets are dried on suspended wire gauze, while protected from air currents, the final drying being effected under pressure. Strips about 7 inches long, 0.1 inch thick, and 0.33 inch wide, are then cut from these, and the centre portion (about 2.5 inches) ground on a grinding wheel. The strips are varied in thickness and width in order to check the results. The tensile strength of strips thus prepared is determined by means of a Schopper tensile machine. Samples of three different commercial glues thus examined gave the following average results: (1) 13.240 lbs.; (2) 8.523 lbs.; and (3) 11.573 lbs. per square inch.

C. A. M.

Estimation of Trimethyleneglycol in Distilled Glycerol. C. A. Rojahn. (*Zeitsch. anal. Chem.*, 1919, 58, 433-442).—The quantity of trimethyleneglycol may be calculated from the sp. gr. and water content of the sample. Glycerol has a sp. gr. of 1.2653 at 15°/15° C., whilst that of the glycol is 1.0573. The sp. gr. of glycerol is diminished by the presence of the glycol or water, or both, but as the

quantity of water is known (by estimation), the percentage of the glycol is found by simple calculation. Tables and graphs are given in the original to facilitate the calculation. To estimate the water, about 2 grms. of the sample are placed on asbestos and dried for twenty-four to forty-eight hours over phosphorus pentoxide at a pressure of about 10 mm.

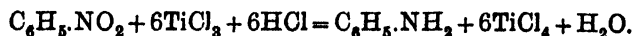
W. P. S.

Detection of Methyl Alcohol in Spirits. P. Hasse. (*Pharm. Zentr.*, 1920, 61, 177-182; through *J. Soc. Chem. Ind.*, 1920, 39, 345 A.)—The sample is distilled and 0.5 c.c. of the distillate (containing not more than 0.025 c.c. of alcohol) is mixed with 1 c.c. of 5 per cent. potassium permanganate solution, 2.5 c.c. of dilute sulphuric acid (sulphuric acid, 19 grms., water 200 c.c.), and, after standing two minutes, the mixture is decolorised by the addition of 1 c.c. of 10 per cent. oxalic acid solution. To 0.5 c.c. of this mixture are then added 1 drop of peptone solution (= 2.5 mgrms. of peptone), and 1 c.c. of sulphuric acid containing iron (0.05 gm. of iron alum dissolved in 1 c.c. of water and added to 300 grms. of sulphuric acid). A deep blue colour is obtained if the spirit contained 1 per cent. of methyl alcohol; a red-blue colour is produced by 0.3 per cent. of the alcohol. Pure ethyl alcohol gives a yellowish-red coloration with the test. If an indication of the presence of methyl alcohol is obtained, it should be confirmed by the morphine and magenta-sulphurous acid tests.

Estimation of Mineral Constituents in Organic Substances, especially those containing Phosphorus. J. Grossfeld. (*Chem. Zeit.*, 1920, 44, 285-286.)—Substances such as albumin, yolk of egg, blood, etc., can be burnt readily to a white ash if they are treated previously with magnesium acetate solution and dried. The whole of the phosphorus present in the substance is retained in the ash. The magnesium acetate solution is prepared by dissolving 50 grms. of magnesia in a slight excess of acetic acid and diluting the solution to 1 litre; 20 c.c. are sufficient for 5 grms. of organic substance. In the estimation of ash constituents an allowance must of course be made for the amount of magnesia added as acetate.

W. P. S.

Volumetric Estimation of Nitro-Aromatic Derivatives. D. Florentin and H. Vandenberghe. (*Bull. Soc. Chim.*, 1920, 27, 158-166.)—Contrary to various published statements, the reduction of nitrotoluenes by stannous chloride gives, in addition to pure toluidines, *p*-chlorotoluidines, owing to the transposition of hydroxylamines which are formed as intermediate products. Hence only two molecules of stannous chloride react instead of the three required by the normal reaction. For this reason the volumetric estimation of nitrotoluenes by means of stannous chloride never gives correct results, and is especially inaccurate in the case of *o*-nitrotoluene. Other nitro derivatives of benzene may be quantitatively estimated by titration with stannous chloride in the presence of hydrochloric acid. With the exception of *o*-nitrotoluene, titanous chloride reduces nitro derivatives of benzene quantitatively in accordance with the equation:



The reduction may be effected by heating 0.2 gm. of the nitro derivative with 10 c.c. of standardised titanous chloride solution in a sealed tube for two hours, and, after cooling, diluting the liquid to 100 c.c., shaking thoroughly, and running it from a burette into a known quantity of ferric sulphate solution, using thiocyanate as indicator. Polynitro derivatives may be titrated in an open flask in presence of a current of carbon dioxide. In the case of *o*-nitrotoluene the error does not exceed 3 per cent.

C. A. M.

Estimation of the Nitro Group in Aromatic Organic Compounds. T. Callan, J. A. R. Henderson, and N. Strafford. (*J. Soc. Chem. Ind.*, 1920, 39, 86-88r.)—The usual method for the estimation of nitro groups in aromatic compounds is by reduction with titanous or stannous chloride and titration of the excess of reducing agent. The method of Knecht and Hibbert, using titanous chloride, gives accurate results in most cases, but certain substances, such as *o*-nitroanisole, nitrocresyl methyl ether, α -mononitronaphthalene, and others, only give about two-thirds of their theoretical value. This is particularly the case when the reduction is effected in the presence of alcohol. The authors show that the low results are due to the simultaneous chlorination of the aromatic compound, and that the nitro derivatives yield chlorinated amines. Nearly normal values are obtained if chlorination be impeded by previous sulphonation of the nitro compound, and the reaction is perfectly satisfactory if titanous sulphate be used for the reduction instead of titanous chloride. The only objection to titanous sulphate is its extreme sensitiveness to atmospheric oxidation at the boiling temperature. It is necessary, therefore, to boil the titanous sulphate solution in a flask provided with a two-holed stopper with narrow entrance and exit tubes for carbon dioxide, and to work with great care to prevent oxidation; the liquid is cooled in a stream of carbon dioxide, and may then be titrated with a standard solution of iron alum, no special precautions being necessary with the cold solution. The reducing agent is prepared by adding 400 c.c. of commercial titanous sulphate (about 12 per cent.) to about 500 c.c. of dilute sulphuric acid (1 in 4), boiling for a few minutes, cooling, and making up to 1,000 c.c. The use of stannous chloride as a reducing agent offers no advantages over that of titanous chloride, the tendency to chlorination being at least as great. A very useful method, though of limited application, consists in reducing the nitro compound and titrating the amine with nitrous acid. About $\frac{1}{20}$ gm. mol. of the nitro compound is dissolved or suspended in excess of dilute hydrochloric acid, and zinc dust is added gradually in considerable excess while the solution is warmed. After about half an hour the solution is filtered, diluted, cooled by the addition of ice, and titrated with $\frac{N}{2}$ sodium nitrite until a drop of the liquid on iodide-starch paper indicates an excess of nitrous acid. In this reaction the formation of chlorinated amines is immaterial provided the products of the reduction form diazo compounds with nitrous acid. It is important that the acid and the zinc dust be free from iron, or, alternatively, a blank determination must be made to ascertain the amount of nitrite consumed by reaction with the ferrous iron present.

J. F. B.

Estimation of Incompletely Nitrated Phenol in the Mother-Liquors of Melinite. Marquoyrol and P. Carré. (*Bull. Soc. Chim.*, 1920, 27, 127-140.)—The volumetric estimation of incompletely nitrated phenol in the mother-liquors of melinite by means of bromine invariably gives too high results, the error varying with the composition of the sample and with the conditions of the nitration. This is due to the fact that the incompletely nitrated phenol is present in various forms which behave differently towards bromine. There are also always present two isomeric sodium dinitrophenol-sulphonates, and sometimes sodium mononitrophenol-sulphonates, each of which reacts differently with bromine according to the conditions of temperature, duration of contact, etc. Moreover, the bromine also reacts with the residual picric acid in the liquid. Results sufficiently accurate are obtained by completing the nitration of the mother-liquids and titrating the picric acid formed: 100 c.c. of the sample are concentrated until their b.-pt. reaches 125° C., and are then heated with 25 c.c. of nitric acid (62 per cent.) for fifteen to twenty minutes at 115° to 120° C., after which an additional 10 c.c. of acid are added, and the heating continued until the b.-pt. again reaches 120° to 125° C. The liquid is then diluted to 100 c.c. with cold water, and the picric acid separated with the aid of a filter pump, washed five times with 4 c.c. of water and titrated with $\frac{N}{10}$ sodium hydroxide solution with methyl red as indicator. A correction of 0.12 to 0.16 gram. (according to the amount of picric acid) is made for the solubility of the picric acid in the washing water. C. A. M.

Determination of Cellulose in Rubber Goods. S. W. Epstein and R. L. Moore. (*Indiarubber J.*, 1920, 59, 559-566; through *J. Soc. Chem. Ind.*, 1920, 39, 343A.)—One half gram. of the rubber is digested with 25 c.c. of freshly distilled cresol (b.-pt. 198° C.) for four hours at 160° to 185° C., then cooled and 200 c.c. of light petroleum spirit (b.-pt. 45° to 50° C.) added with constant agitation. After allowing to settle and decanting the liquid through a Gooch crucible containing a pad of acid-treated, ignited asbestos, the residue is washed thrice with petroleum spirit, five times with hot benzene, and once or twice with acetone; it is then treated with hot hydrochloric acid (10 per cent.), transferred completely to the crucible, washed ten times with hydrochloric acid, then with water until free from chlorides, and treated with acetone and subsequently with a mixture of acetone and carbon bisulphide (1 : 1) until the extracts are colourless. The residue is washed with alcohol, dried for ninety minutes at 105° C., transferred to a weighing bottle and weighed. The extracted material is digested in a beaker with 15 c.c. of acetic anhydride and 0.5 c.c. of sulphuric acid for thirty minutes at 75° C., cooled, treated with 25 c.c. of 90 per cent. acetic acid, filtered slowly through a pad of treated asbestos in a Gooch crucible, washed repeatedly with hot 90 per cent. acetic acid and then five times with acetone. The crucible is placed in a weighing bottle, dried for two hours at 150° C., and then weighed. The loss in weight on acetylation represents the cellulose. The presence of leather does not interfere with the method, but in such a case it is desirable to digest with cresol at 120° C. for sixteen hours. The acetylation process indicates 95 per cent. of any wood present, 90 per cent. of any jute, 21 per cent. of cork, and 70 per cent. of any leather.

INORGANIC ANALYSIS.

Use of Cupferron in Quantitative Analysis. G. E. F. Lundell and H. B. Knowles. (*J. Ind. and Eng. Chem.*, 1920, 12, 344-350.)—From an experimental study of the method of estimating metals by precipitation with cupferron (the ammonium salt of nitrosophenylhydroxylamine) the following conclusions have been drawn: The reagent can be used for the accurate estimation of iron, copper, titanium, zirconium, thorium, and vanadium. In any given estimation the partial or complete precipitation of these metals must therefore be taken into consideration, whilst the following elements also interfere with the estimation of other metals: Lead, silver, mercury, tin, bismuth, cerium, thorium, tungsten, uranium in the quadrivalent condition, and vanadium; and in certain cases phosphorus, alkali salts, and alkaline earth compounds if present in excessive amounts. Hence the cupferron method is only applicable when the qualitative composition of the material is known. The reagent may be advantageously used for certain separations, such as iron from manganese, and titanium from aluminium and manganese. C. A. M.

Analysis of Iron Ore. E. Little and W. L. Hult. (*J. Ind. and Eng. Chem.*, 1920, 12, 269-273.)—The use of dichromate as an oxidising agent instead of permanganate is recommended, particularly since it has no action on dilute hydrochloric acid. Up to now the principal objection has been the absence of a convenient indicator. Potassium iodide and starch may, however, be used, provided the influence of the ferric ion be inhibited by the introduction of ammonium fluoride. An excess of dichromate is used, and the liberated iodine is titrated back with thiosulphate. The weight of ore in the solution to be titrated should contain iron equivalent to about 25 c.c. of a $\frac{N}{10}$ solution. The ore should be dissolved in 20 to 35 c.c. of hydrochloric acid 1:1; an excess of acid produces an indefinite end point. The amount of potassium iodide should be 5 grms. For the reduction of the iron solution a slow passage through the Jones reductor gives the most satisfactory results; fairly good results can be obtained with stannous chloride using mercuric chloride to destroy the excess of the latter, but, with any appreciable excess of stannous chloride, difficulties are experienced owing to the reaction of the iodine with the calomel produced. The amount of ammonium fluoride used should be about 5 grms. The total volume after titration should be 250 to 350 c.c. J. F. B.

Volumetric Estimation of Manganese. P. Nicolardot, A. Reglade, and M. Geloso. (*Comptes rend.*, 1920, 170, 808-810.)—In the estimation of manganese by Knorre's method (precipitating the manganese as dioxide by ammonium persulphate, dissolving the precipitate in excess of ferrous sulphate solution and titrating the excess with permanganate solution) the reaction proceeds according to the equation: $\text{MnO}_2 + 2\text{FeO} + 4\text{H}_2\text{SO}_4 = \text{Fe}_2(\text{SO}_4)_3 + \text{MnSO}_4 + 4\text{H}_2\text{O}$, and Fe should be equivalent to 0.4917 Mn. It is found in practice that a higher factor is required, and different workers have suggested values ranging from 0.392 to 0.501. The authors find that the presence of iron affects the composition of the manganese dioxide preci-

pitae. In the absence of iron, the factor is 0.498, whilst with 40 per cent. of iron (calculated on the manganese-iron mixture) the factor is 0.4929. Dilution is without influence on the results.

W. P. S.

Estimation of Mercury by Gluckmann's Method. A. Abelman. (*Zeitsch. anal. Chem.*, 1919, **58**, 443-445.)—Gluckmann's method for the estimation of mercury (precipitation with an excess of oxalic acid and subsequent titration of the excess with permanganate solution) yields untrustworthy results owing to the solubility of mercuric oxalate and to the formation of basic salts. The error due to solubility may be decreased by adding potassium nitrate, whilst the addition of a small quantity of nitric acid prevents the production of basic salts during the precipitation. The mercury salt solution is treated with 3 c.c. of 5N nitric acid and a large excess of oxalic acid, 50 c.c. of saturated potassium nitrate solution are then added, the mixture diluted to 100 c.c., filtered, and the excess of oxalic acid is titrated in the filtrate. When thus modified, the method yields from 98.6 to 100 per cent. of the mercury present.

W. P. S.

Estimation of Potassium as Perchlorate. G. P. Baxter and M. Kobayashi. (*J. Amer. Chem. Soc.*, 1920, **42**, 735-742.)—When much sodium perchlorate is present with the potassium perchlorate, the sodium salt should be removed by washing with alcohol containing perchloric acid before the washing with alcohol saturated with potassium perchlorate is commenced (*cf.* ANALYST, 1917, **42**, 155). The authors agree with Gooch and Blake (*ibid.*, 1918, **43**, 277) that sodium perchlorate causes the precipitation of potassium perchlorate from the saturated alcoholic solution of the latter, particularly when in contact with solid potassium perchlorate. The washing should be carried out at 0° C.

W. P. S.

Reaction of Potassium Anhydrotellurate. P. Hulot. (*Bull. Soc. Chim.*, 1920, **27**, 33.)—On fusing tellurium, or tellurous acid or alkali tellurites with potassium nitrate, there is not complete conversion into potassium tellurate, but a white powder is left as a residue which is insoluble in water and in hot concentrated mineral acids. This product is potassium anhydrotellurate, $\text{Te}_4\text{O}_{13}\text{K}_2$. It may be reduced to metallic tellurium by suspending it in water containing a third of its volume of hydrochloric acid and adding a few fragments of zinc. The reaction is applicable to the treatment of tellurium ores, which may thus be decomposed by the wet method without loss of tellurium.

C. A. M.

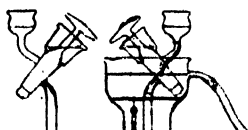
Potassium Chlorate as a Standardising Substance for Alkali Solutions. H. B. Van Valkenburgh. (*J. Amer. Chem. Soc.*, 1920, **42**, 757-760.)—Potassium chlorate, recrystallised and dried for five hours at 240° C., may be used for standardising alkali solutions. A weighed portion of the dry salt is dissolved in water, the solution boiled for ten minutes, a current of sulphur dioxide is then passed into the boiling solution for thirty minutes, or until all the chlorate is reduced, the boiling continued for about five minutes, and the resulting sulphuric acid then titrated with the alkali solution, using phenolphthalein as indicator. The reaction is expressed by the equation: $\text{KClO}_3 + 3\text{SO}_2 + 3\text{H}_2\text{O} = 3\text{H}_2\text{SO}_4 + \text{KCl}$.

W. P. S.

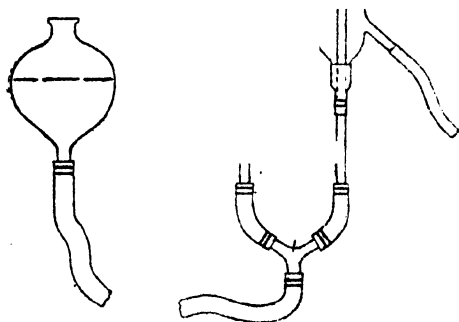
Potassium Hydrogen Phthalate as a Standard in Volumetric Analysis. W. S. Hendrixson. (*J. Amer. Chem. Soc.*, 1920, **42**, 724-727.)—Further experience with potassium hydrogen phthalate, recommended previously by the author (*ANALYST*, 1915, **40**, 509) as a standard substance for volumetric solutions, shows that the salt can be prepared readily from phthalic anhydride and a slight excess of potassium carbonate, and that, after two recrystallisations, it is pure enough for all practical purposes. The salt is free from water of crystallisation and is not hygroscopic.

W. P. S.

APPARATUS, ETC.



Instrument for Measuring Vapour Tension. H. Moore. (*J. Soc. Chem. Ind.*, 1920, **39**, 78-80t.)—The apparatus shown in the diagram has been primarily devised for determining the vapour tension and, consequently, the volatility of motor spirit. It consists of two tubes of about $\frac{1}{4}$ inch bore and about 800 mm. in length, connected with each other and with a levelling bulb by means of a Y-piece and thick-walled rubber tubing. The two tubes have cocks at the top sealed with mercury, and one of them is surrounded by a water-jacket in which a thermometer is suspended. In making a determination, mercury is introduced into the two cups attached to the cocks, which are opened while the levelling bulb is raised. The cocks are then closed, and the levelling bulb lowered, so that two barometer tubes are formed. One cup is filled with motor spirit, and a small amount, measured by a mark below the cock, is run into the tube, and sufficient mercury afterwards placed in the cup to form a seal. On lowering the levelling bulb the barometric height may be read on the empty tube, whilst the jacketed tube, containing the motor spirit, gives a reading which is the barometric height less the vapour pressure of the motor spirit at the temperature of the determination. The difference in the heights of mercury in the two tubes is the vapour-pressure reading.



For accurate readings a correction for the density of the mercury is necessary, but in the case of the motor-spirit tests the amount is less than the normal experimental error.

C. A. M.

Barger's Microscopical Method of determining Molecular Weights. Part I. Kumao Yamakami. (*J. Biol. Chem.*, 1920, **41**, 103-113.)—Barger's method for determining molecular weights (*J. Chem. Soc.*, 1904, **85**, 286) is stated by the author to be based upon vapour pressure. Series of drops, taken from two solutions, the one the standard, the other a given solution, of which the molecular weight of the solute is to be determined, are alternately introduced into a capillary tube, and the change in the length of the drops is studied by means of a micrometer. If one series of drops is observed to have increased while those alternating with them have decreased, it is to be assumed that the solution from which the former series of drops was taken has a higher molecular concentration than the latter. The given solution is thus compared with a series of standard solutions of known molecular concentrations, and in this way two limits are reached for the unknown molecular concentration. In a large number of experiments described by the author, he concludes that it is impossible to explain the reactions involved without assuming that the drop-change occurred because of a transference, not only of vapour, but also of liquid from the weaker solution through the film furnished by the liquid membrane which extended between the two drops. The author considers that he has proved that the force which effects the volume-change of the drops is not simply the vapour pressure, but also the osmosis through the thin film of liquid between the drops. If the length of the intervals between the drops is 2-3 mm., $\frac{5}{8}$ to $\frac{9}{7}$ of the volume-change of the drops is attributable to osmosis. Further, as osmosis as well as the lowering of vapour pressure are equally the results of one and the same internal energy of solution—that is, the molecular and ionic attraction of solute for solvent, which is of the same magnitude for all kinds of molecules and ions in the same solvent at the same temperature—it is possible to measure molecular weights or the same degree of association or dissociation by investigating the volume-change of drops which are brought in contact with drops of standard solutions of known molecular concentrations.

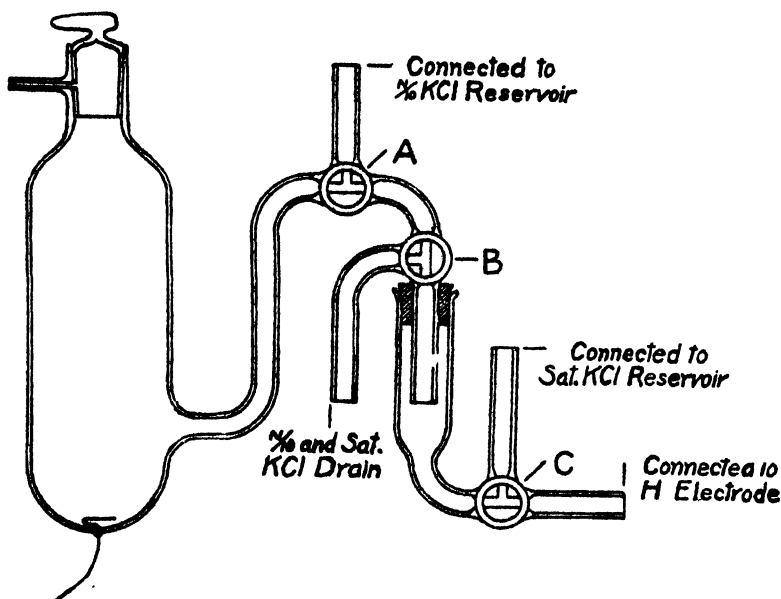
H. F. E. H.

Colorimetric Determination of Titration Curves without Buffer Mixtures. L. J. Gillespie. (*J. Amer. Chem. Soc.*, 1920, **42**, 742-748.)—The hydrogen ion exponents are determined by the use of a colour standard consisting of a pair of test-tubes containing together 10 drops of indicator solution of suitable strength, the drop ratio being varied from 1 : 9 to 9 : 1. One of the tubes contains dilute alkali solution and the other dilute acid. Reference to a table gives the hydrogen ion exponent corresponding with the drop ratio, or the exponents can be calculated from the relation $P_H = k + \log (\text{drop ratio})$. Where the logarithms are common, the drop ratio is the ratio of the number of drops of the indicator solution in the alkaline tube to the number in the acid tube, and the values of k are as follows: Tetrabromophenol-sulphonphthalein, 4.1; methyl red, 5.0; dibromo-*o*-cresol-sulphonphthalein, 6.3; dibromothymol-sulphonphthalein, 7.1; phenol-sulphonphthalein, 7.7; *o*-cresol-sulphonphthalein, 8.1; thymol-sulphonphthalein, 8.8.

W. P. S.

New Calomel Electrode. A. E. Koehler. (*J. Biol. Chem.*, 1920, **41**, 619-620.)—The apparatus is arranged in such a manner that each of the potassium

chloride solutions employed, when mixed after use, may be readily rinsed out and replaced by fresh solution. The $\frac{N}{10}$ potassium chloride occupies the left side of the apparatus as far as stopcock *B*, while the remainder is filled with the saturated

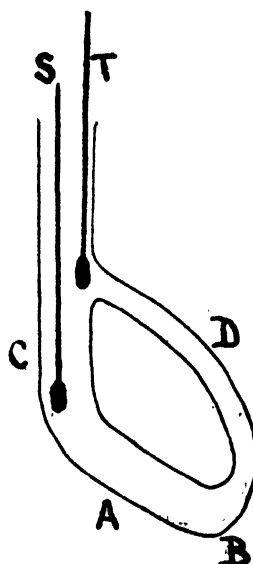


solution. Contact is made between the two solutions around *B* which is not greased. After use, the tube extending from *A* to *B* is washed out with $\frac{N}{10}$ potassium chloride solution from the reservoir, and, similarly, the section from *B* to *C* is rinsed with the saturated solution by suitable manipulation of the stopcocks. During a period of two months' continual use the E.M.F. of the electrode remained constant.

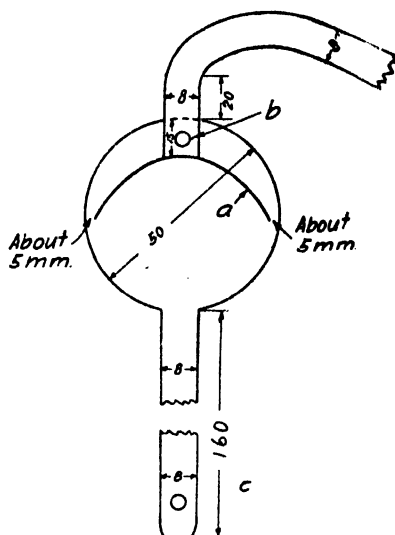
T. J. W.

Apparatus for the Determination of Melting-Points. L. M. Dennis. (*J. Ind. and Eng. Chem.*, 1920, 12, 366-368.)—In determining the melting-points of substances in capillary tubes attached to a thermometer and immersed in pure concentrated sulphuric acid, the accuracy of the results is greatly increased by using a tube of the form shown in the diagram for the sulphuric acid bath. This accelerates the circulation of the acid and thus equalises its temperature, as may be demonstrated by means of thermometers *S* and *T* fixed in the positions shown. The difference between the temperatures recorded by the two thermometers is much smaller, even when the tube is rapidly heated, than when a Thiele tube is employed and heated more gradually.

C. A. M.

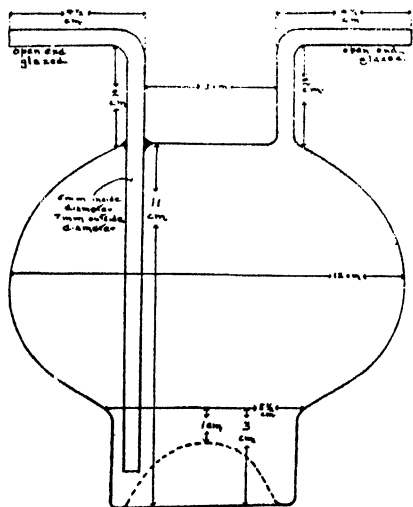


Distillation Connecting Bulb. C. M. Clark. (*J. Ind. and Eng. Chem.*, 1920, 12, 366.) The bulb shown in the diagram has been designed to prevent spray from



being carried over into the distillate. This is effected by means of a baffle plate, *a*, in the form of a deep watch-glass, which extends to within about 5 mm. of the sides of the bulb, and prevents any spray from reaching the exit holes at *b*. C. A. M.

Condenser for the Determination of Crude Fibre. C. A. Clemens. (*J. Ind. and Eng. Chem.*, 1920, 12, 288-289.)—A condenser has been designed to



rest inside the top of a lipless beaker, such as is used in digesting, for the estimation of crude fibre. It consists of a glass bulb (see fig.), one side of which is blown in such a manner as to extend downward. The lower surface of this projection is concave; thus the bottom is ring-shaped, and furnishes a suitable surface upon which the apparatus may rest when not in use. Into the top part of the bulb two glass tubes are sealed, one serving as an inlet and the other as an outlet for a stream of cold water. The condenser may be supported by attaching a wire to the inlet and outlet tubes, and fastening it to an iron rod clamped to a ring stand; the beaker can then be removed without disturbing the condenser. Owing to the concave

surface of the effective portion of the condenser, an increased cooling area is obtained, and foam is prevented from rising to the top of the beaker. The condenser may be used with lipless beakers ranging in size from 500 to 1,000 c.c. J. F. B.

REPORTS.

Report on the Work of Inspectors of Foods for the Year 1918-19.

A. W. J. Macfadden. (Extract from the Annual Report of the Medical Department of the Local Government Board for 1918-19. H.M. Stationery Office, 3d. net.)

WORK OF INSPECTORS OF FOODS.—During the period covered by the report it has been found necessary to devote much attention to the inspection of imported meat. A considerable proportion of the meat was of very poor quality, and some of it had been imported from regions where stock is used chiefly for draught purposes, and feeding for market purposes is practically unknown. Such meat had seldom been seen in this country in the past, and to those unfamiliar with its appearance is often repulsive, and suggestive of wasting disease. A good deal of the meat, too, had suffered from rough handling and conveyance in unsuitable waggons. The surface was often dirty, and, in places, had become slimy or stale owing to exposure. These conditions were explained as having been due to difficulties in obtaining suitable labour and rolling stock for the conveyance of the meat.

ARSENIC IN FOOD MATERIALS.—The steps taken by manufacturers of baking-powder materials in this country to safeguard their products from arsenical contamination appear to have been successful. It is desirable, however, that public analysts throughout the country should continue to keep food materials of this class under observation locally.

MATTERS CALLING FOR REFORM. (1) *Inspection of Home-Killed Meat.*—The question of the protection of the public from dangers due to the consumption of meat derived from diseased animals is one which the author considers still requires very much more attention than it has so far received. He discusses at considerable length the arrangements that have already been made for the provision of public abattoirs and the difficulties which have arisen owing to the continued preference of many butchers for old and unsuitable slaughter-houses. Until the Ministry of Food took over the control of the sale and slaughter of animals for food there was a large trade done in various parts of the country by unscrupulous dealers, who carried on a highly lucrative business in the trading of diseased and emaciated animals for surreptitious conversion into meat, and no system of local meat inspection, however good, would serve to protect consumers against diseased meat in districts in which inspection is absent or inefficient. There is only one method of ensuring the freedom of meat from disease, and that is by careful examination of the carcass at the time of slaughter, when all viscera are available for inspection. The only practicable means by which this can be brought about is by requiring all animals intended for food purposes to be brought to a public abattoir and by providing skilled inspectors in sufficient numbers to inspect thoroughly, both before and after slaughter, every animal brought there. This would result in the closing of all private slaughter-houses as such, and, in order to be of practical advantage to public health, the requirements would need to operate in town and country alike. Their

application to urban areas alone would not be sufficient, as this would merely accentuate past evils by driving the trade in diseased animals exclusively into rural areas where there is no inspection at all. The position in regard to this matter is, from the public health point of view, most unsatisfactory. The difficulties in the way of dealing with it in the past have been very great, but the opposition to public abattoirs which has hitherto been encountered from butchers may possibly be modified in future on account of the altered conditions which have resulted under the Ministry of Food's restrictions as to slaughtering.

(2) *Supervision of Places where Food is Prepared or Kept for Sale for Human Consumption.*—The powers which local authorities possess under the Public Health Acts are inadequate for securing such a standard of suitability and cleanliness of premises, and of care in the materials used and processes employed in factories and other places where food is prepared or kept for sale, as is necessary to maintain conditions in these places which would be reasonably safe from the point of view of consumers of the materials manufactured there. The nuisance clauses of the Public Health Act and the procedure laid down in the Act for dealing with nuisances are quite unsuitable for controlling such places. Very unsavoury details of the foul conditions existing in many places where pies, sausages, and other foods are prepared for sale are described in the report, and it is stated that it has become a matter of increasing urgency that the regulation of this class of premises should be undertaken. The report closes with some general remarks upon problems in human nutrition, more particularly in connection with the accessory factors or "vitamines," concerning which there has been so much recent discussion. Processes of manufacture and the manner in which food is prepared may exert a profound influence upon its ultimate vitamine content. The antiscorbutic vitamine of fresh vegetables is destroyed by drying or by prolonged cooking, and the antineuritic vitamine in foodstuffs, though more stable, is affected injuriously by exposure to high temperatures, such as may be employed in the sterilisation of foods. Many proprietary foods and invalid foods appear to be practically devoid of vitamins. Organised research on the subject of accessory food factors, or vitamins, is already in progress in this country. Other aspects of the problem of nutrition, especially those relating to the economic situation, are, however, in urgent need of research work properly organised and directed on systematic lines.

H. F. E. H.

Food Values and Dairy Products. O. R. Overman. (Circular No. 235, Agric. Exper. Sta., University of Illinois.)—The circular deals very fully in a statistical manner with the food values of all the more usual dairy products, numerous tables of chemical composition, calorie value and cost per calorie being included in the work. The cheapness of dairy products as regards both protein and total energy is emphasised, and stress is laid upon the advantage which would accrue from the standpoint of economy if greater use were made of them by purchasers of flesh and other foods. Emphasis is also laid upon the fact that milk containing 4 per cent. fat is a more economical source of both protein and energy than are meat, eggs, poultry or fried fish, while even skim milk is a far cheaper source

of protein and energy than are other foods of animal origin, and, considered only as a source of protein, it is more economical than dried peas or beans. Comparisons on the energy basis alone are, it is pointed out, in many cases unfair to one or other of the foods compared, and it is advisable always to bear in mind that a better way to look at the problem is from the point of view of the number of calories that can be bought for a given sum, and, from the tables appended tabulating this information, it becomes very evident that milk is relatively inexpensive as compared with many other foods commonly considered to be much more nutritious.

H. F. E. H.



REVIEW.

LEGAL CHEMISTRY AND SCIENTIFIC CRIMINAL INVESTIGATION. By A. LUCAS. Pp. viii + 181. London: Edward Arnold. Price 10s. 6d. net.

Every analyst in general practice has occasionally to solve problems of a legal character, and in such cases will generally find that he can obtain little assistance from ordinary textbooks. Usually he has to devise his own methods, and to test them by the results of experiments upon known materials under artificial conditions. As director of the Government Laboratory in Cairo, the author of this book has been exceptionally placed for gaining experience in connection with legal and criminal cases in which chemical methods have been required, and he has embodied his notes on these cases under a series of classified headings. The precedent might well be followed, and if other chemists would publish notes upon the methods used in similar out-of-the-way cases a literature dealing with forensic chemistry (as distinct from toxicology) would eventually be available.

The subjects dealt with are arranged in alphabetical order for easy reference, and cover a great deal of ground, since they practically touch upon most branches of criminal activity, as will be gathered from the headings of some of the sections. These include subjects as remote as forged antiquities, examination of bullets, hashish, damage to crops, and finger-prints. In many of the cases no original methods of examination have been devised, but microscopical and chemical methods have been adapted to the special end in view. In other cases, however, the author gives interesting details of his investigation in connection with special subjects, such as the disintegration of building-stones in Egypt, and the nature of the material used for embalming mummies. At the end of each section references are given to the literature which has a bearing upon questions likely to arise in connection with the subject. The section to which most space has been given is that dealing with the examination of documents, and this is full of interesting information as to the use of early inks and papers in Egypt. The advantage of having authentic information as to the dates of changes in the composition of writing materials is shown in several of the instances cited, and in particular in connection with forgeries where an attempt has been made to imitate an old ink. In one case mentioned all but five

out of 168 documents were falsified in all manner of ways, from the simple forgery of a signature to the fabrication of the entire document. The value of chemical and microscopical examination is also made evident in connection with the cognate subjects of tampering with letters in transit and the fabrication of seals. The sections dealing with alcoholic liquids and with food and drugs are perhaps a little out of place as they stand, since much of the information (apart from details concerning Egyptian products) will be familiar to most analytical chemists. The same criticism may also be applied to the section on the pollution of water by sewage, and, to bring these two sections within the general scheme, details of exceptional cases within the author's experience should be given.

This, however, is a minor criticism, and, regarded as a whole, the book may be strongly recommended to all chemists whose work is likely to include legal questions. It is to be hoped that the author may see his way to expand his notes into a general textbook of forensic chemistry.

C. A. MITCHELL.



THE INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

PASS LIST.

APRIL (1920) EXAMINATIONS.

THE results of the examinations of the Institute recently held in London, Manchester, and Sheffield have now been published. The following candidates were successful :

For the Fellowship: In the branch of Organic Chemistry, R. O. Eames, B.Sc. (Wales), and H. W. B. Clewer; in the branch of the Chemistry of Food and Drugs, Fertilisers and Feeding-Subs, Soils and Water, Norman Ratcliffe; in the branch of the Chemical Technology of Textile Manufacture, E. Clayton.

For the Associateship: In the branch of Organic Chemistry, E. C. Pickering, B.Sc. (Lond.), and S. B. Phillips; in the branch of the Chemistry of Food and Drugs, Fertilisers and Feeding-Subs, Soils and Water, F. N. Appleyard and A. Lees; in the branch of the Chemical Technology of Textile Manufacture, R. Humphries; in the branch of the Chemical Technology of Cement Manufacture, S. Bowman.

For the Certificate: In the branch of the Chemistry of Food and Drugs, Fertilisers and Feeding-Subs, Soils and Water, S. Dixon, M.Sc., A.I.C.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

ORDINARY MEETING, JUNE 2, 1920.

HELD at the Chemical Society's Rooms, Burlington House, Mr. Alfred Smetham, President, in the chair.

A certificate was read for the first time in favour of Mr. T. K. Ghose, B.A.

A certificate was read for the second time in favour of Mr. Udolphus Aylmer Coates.

The following were elected Members of the Society: Misses Dorothy Gertrude Hewer, B.Sc. (London), Rita Catherine Hawkins Johnson, Messrs. Harold Hall, F.I.C., Geoffrey Trelawney Bray, A.I.C., Frank William George King, and John Herbert Stubbs, M.Sc. (Victoria), F.I.C.

The following papers were read: "The Estimation of Nitroglycerine," by H. Droop Richmond, F.I.C.; "The Perchlorate Method for Potash," by R. Leitch Morris, F.I.C.; "Apparatus for Evolution Methods of Analysis: An Improved Form of U-Tube," by E. R. Dovey, A.R.C.S., A.I.C.

* * * * *

ESTIMATION OF THE AGE OF INK IN WRITING.

By C. AINSWORTH MITCHELL, M.A., F.I.C.

(Read at the Meeting, May 5, 1920.)

QUESTIONS as to the age of ink in writing are frequently raised in connection with the examination of documents. Sometimes, for instance, there may be reason for doubting whether an entire document is as old as it purports to be, or information may be wanted as to whether the whole of the writing was written at the same time, or, if not, which portion was written first. In many cases the problem is insoluble, but in others the facts enable a definite opinion to be expressed, and may even afford convincing evidence.

Anachronisms in the composition of the ink in the writing are among the most

conclusive of such facts, and it is, therefore, a matter of considerable importance to know the approximate dates at which such changes have occurred.

EVOLUTION OF WRITING INKS.

The writing ink of the Greeks and Romans, like some of the modern inks still used in the East at the present day, was a mixture of fine lamp-black or other form of carbon with a dilute solution of gum or glue. It is not possible to state the exact period when carbon inks of this type were replaced by iron-gall inks. The earliest reference made to the latter by the monk Theophilus is in an encyclopædia of Christian art of the eleventh century, and it is probable that by the end of the thirteenth century the modern type of ink had practically replaced the ancient inks.

Originally the modern inks were simply prepared by mixing an infusion of galls with iron sulphate and gum, and were allowed to undergo considerable oxidation before being used.

Logwood was used in the eighteenth century to a limited extent to intensify the colour of iron-gall inks (Ribeaucourt, *Ann. de Chim.*, 1792, 15, 113), whilst the use of indigo for the same purpose is mentioned by Eisler (Dintefass, 1770), and was adopted in this country in 1836 (*Mechanics' Magazine*, 1836, 25, 229). The use of aniline dyestuffs was first claimed in England by Croc (Eng. Pat., 2,972, 1861). I have the authority of Mr. W. Baker, of Messrs. Mabie, Todd and Co., for stating that aniline blue dyes of different kinds were commonly employed in the manufacture of blue-black ink upwards of forty years ago. In my own experience I have been able to trace a blue pigment in writing in old ledgers for about thirty-five years, the writing prior to that date in the same ledgers being in iron-gall inks which did not show any indication of blue pigment.

This point has been found of great importance on several occasions in connection with fraudulent claims for old age pensions. For example, in support of the claim family Bibles have been produced in which entries supposed to be seventy years old have been found to be written in ink containing an aniline dye.

In the case of *Rex v. Menzies*, tried in 1916, the defendant was accused of having uttered fabricated documents dating from 1719 to 1772 in support of his claim to a baronetcy. Certain alterations in these documents were alleged to have been written subsequently to 1914, a mixture of blue-black ink and an aniline red ink having, it was stated, been used for this purpose. Evidence was given that the ink in this writing did not contain aniline dyes, and that old ink had, therefore, not been imitated in the manner stated. As there was no evidence that the alterations in the documents had been made subsequently to 1914, when the defendant had acquired them, he was acquitted.

An earlier case of a similar kind is cited by Wills in his "Circumstantial Evidence," p. 142, in which Alexander Humphreys was tried at Edinburgh in 1839 for forging several documents in support of his claim to the Earldom of Stirling. On the margin of one of these documents, dated 1639, lines were drawn in red ink, but it was officially proved that such lines were not introduced into the Chancery Office until 1780. Evidence was also given that the ink in some of the attestations

was a modern ink made to imitate ink which had become old. It would be instructive to know the grounds on which this last conclusion was based.

ARTIFICIAL AGEING OF INK.

In a case in which I was consulted a few years ago there was no doubt as to the artificial ageing of the writing. A cashier confessed that he had falsified certain entries in a ledger, and to make them appear older had written them in a mixture of blue-black ink and Indian ink. I was asked to examine these entries, and found that the writing in question contained carbon particles, which were distinctly visible under the microscope, and that it could not be bleached either by hydrochloric acid or by hypochlorous acid. My results, therefore, confirmed the statement that Indian ink had been used. Incidentally I might mention that the colour of the mixture of Indian and blue-black does not agree very well with that of a naturally aged iron-gall ink.

MICROSCOPICAL DIFFERENCES IN OLD AND RECENT INKS.

For many years after it has been put upon the paper, an ordinary iron-gall ink of the blue-black type will usually have the appearance of a crystalline structure, which appears to be due to pigment attaching itself to the fibres of the paper. Eventually, as the organic matter of the ink decomposes, there is more or less formation of iron oxide or of basic tannates, and the ink not only becomes yellow, but also appears much more amorphous, and in some cases shows isolated patches of darker pigment. Good examples of this are frequently to be seen in writing of the eighteenth century.

As steel pens were not invented until 1808 (Eng. Pat., 3,118), the presence of heavy margins to the lines in writing prior to that date would be a suspicious circumstance. In the case of *Rex v. Menzies*, mentioned before, evidence was given that the additions to a document of 1719 had been made with a steel pen, whereas the rest of the document had the appearance of having been written with a quill pen.

COLOUR OF OLD AND NEW INKS IN WRITING.

When freshly applied to the paper, a mixed solution of iron sulphate and a tannin is pale grey, but it gradually absorbs oxygen from the air, and rapidly darkens as the insoluble tannate forms. In the modern type of blue-black inks containing both the latent blackish pigment and an added dyestuff to give colour pending the formation of the iron tannate, the colour is first bright blue, and rapidly becomes violet as the other pigment is formed.

The completion of this change—i.e., the production of the maximum intensity of colour—will vary greatly with the conditions. The statement which I published some years ago (*ANALYST*, 1908, 33, 80) was correct for the conditions of my experiments, in which thin washes of the inks were applied to paper with a brush and then exposed to a strong light. Under such conditions the maximum colour is reached in about a week, and pure iron tannate then begins to fade, turning yellow

at the edge. Thus, thin washes of inks when exposed to the air underwent the following colour changes on Lovibond's colour scale:

Days.	1.			2.			3.			4.			5.			7.		
	B.	R.	Y.	B.	R.	Y.	B.	R.	Y.	B.	R.	Y.	B.	R.	Y.	B.	R.	Y.
Iron tannate	0.9	0.3	—	1.0	0.5	—	1.3	0.6	—	2.0	0.8	0.2	1.8	0.9	0.3	1.6	0.9	0.3
Blue-black ink	3.0	—	—	3.75	0.5	0.3	4.0	0.7	0.5	4.0	0.7	0.5	3.2	0.7	0.3	3.2	0.6	0.3
Blue-black ink (old) ...	2.1	—	—	2.8	—	—	3.0	0.5	0.5	3.0	0.5	0.5	3.0	0.5	0.5	3.0	0.6	0.5

Days.	10.			17.			23.			28.			35.			43.		
	B.	R.	Y.	B.	R.	Y.	B.	R.	Y.	B.	R.	Y.	B.	R.	Y.	B.	R.	Y.
Iron tannate	1.6	0.9	0.3	1.4	1.0	0.3	1.2	0.9	0.3	1.2	0.9	0.3	1.1	0.9	0.4	1.1	0.9	0.4
Blue-black ink	3.2	0.6	0.3	3.2	0.6	0.3	3.0	0.6	0.2	3.0	0.6	0.2	3.0	0.6	0.2	3.0	0.6	0.2
Blue-black ink (old) ...	3.0	0.7	0.4	2.85	0.7	0.3	2.85	0.7	0.3	2.85	0.7	0.3	2.85	0.7	0.3	2.85	0.7	0.3

If, however, the ink is applied in an ordinary way with a pen, it is left in a deeper layer, and if then kept in a closed book the final intensity of colour may not be reached for upwards of a year.

For example, I found that specimens of writings of the same blue-black ink applied with a pen to the paper at intervals of over a year attained their maximum intensity after a period of eight to ten months. They then had the following colour on Lovibond's colour scale: Blue, 3.6; red, 0.4; yellow, 0.4.

METHODS OF RECORDING THE COLOUR.

It is obvious that if a record be taken of the initial colour of an ink, and the writing is subsequently found to darken rapidly in colour, it is probably comparatively recent, and at any rate will not be more than eighteen months old.

In one case in which I was consulted an ink alleged to be upwards of two years old darkened very materially in a few days, and was thus clearly a forgery.

In the paper which I read before this Society in 1908 I called attention to the use of Lovibond's tintometer for comparing the colour of inks, but did not describe the methods I used. As viewed with the naked eye, the ink in writing on paper is too minute to be satisfactorily examined with the tintometer alone, and so I prepared a series of broad colour stripes with aniline dyes, matched these with the ink in the writing under the microscope, and then took the record with the tintometer from the coloured stripes. This is a cumbersome method, but I have found it useful for demonstrating the colours in court. For most purposes, however, it is much more simple to make a direct comparison between the ink and the tintometer glasses by means of Osborn's comparison microscope, a description of which I gave in a former paper (*ANALYST*, 1917, 42, 3). This instrument has slots in both tubes in which the tintometer glasses can be placed, and the coloured surfaces then appear as two halves of a sphere.

In recording the colour of inks it is sometimes necessary to take into consideration the colour of the paper. For example, a faded brown ink of 1868 gave the following readings: Blue, 0.55; red, 0.7; and yellow, 0.1. This was written on blue paper, which gave a reading of blue 0.50, and evidently contributed to the reading shown by the ink.

Other specimens of old faded inks have given the following readings :

						Blue.	Red.	Yellow.
Ink of 1718	...	...	...	...	...	1·9	1·0	1·9
„ 1792	...	...	...	..	...	—	1·2	0·9
„ 1823	...	...	...	...	...	—	1·1	1·2
„ about 1850	...	...	...	...	...	—	0·6	0·6

I am indebted to Mr. A. W. Johnson, of Messrs. Brown, Johnson and Co., for specimens of writing done with freshly-made ink in the year 1906, before and after the addition of aniline blue, and with and without blotting. The other readings were those given by the specimen of blue-black ink of 1895 (shown on the screen).

	Blue.	Red.	Yellow.
1. Iron-gall ink without aniline blue	2.5	1.4	1.0
2. " " " blotted	1.1	0.4	0.5
3. " with aniline blue	4.0	1.2	0.9
4. " " " blotted	2.0	0.4	0.4
5. Ink in writing of 1895	2.4	1.4	1.1

The ink of No. 5 was originally a well-known bright blue-black ink. It now shows no trace of the original blue dye, and has faded to the colour of No. 1, an ink which contained no aniline blue.

INFLUENCE OF BLOTTING ON THE COLOUR OF INK.

As ordinary blue-black ink contains two pigments, one of which is latent, the effect of blotting is to remove variable quantities of these pigments, and the colour of the ink left on the paper will vary with the length of time before blotting.

In the case of *Rex v. Cohen*, a doctor was accused of "doping" a man to keep him out of the army. He asserted that the man was a regular patient, and as proof thereof produced his books. There were some seven or eight entries of this man's name on various dates, and by a curious coincidence the ink in these were all paler than adjacent entries. Mainly from this circumstance a handwriting expert gave evidence that these entries were open to grave suspicion of forgery, and Dr. Cohen was sentenced to a fine of £100 and six months' imprisonment. He appealed against this sentence, and when I subsequently examined the books I found that there was absolutely nothing to justify any charge of these entries being made subsequently to

adjacent entries. Apart from the many possibilities affecting writing done at different times, the fact that the entries were blotted at different periods, some at the end of a series and sometimes after a single entry, was in itself sufficient to account for variations in the colours and intensity of the inks of adjacent entries.

This is clearly shown in the following series of readings of the colours of ink washes which were applied to the paper for measured periods and then blotted. This colour chart which was shown in court on the hearing of the appeal, proves conclusively that the paleness of the ink in writing is no criterion of the age of the ink, but depends upon the relative proportions of the pigments left upon the paper prior to oxidation.

	B.	R.	Y.	B.	R.	Y.	B.	R.	Y.	B.	R.	Y.	B.	R.	Y.
	5 secs.			10 secs.			15 secs.			30 secs.			90 secs.		
Blue-black ink, faded in bottle	0.3	0.1	—	0.4	0.1	—	0.9	0.1	—	1.0	0.2	0.1	1.5	0.2	0.1
	5 secs.			15 secs.			40 secs.			90 secs.			2 mins.		
Blue-black ink, same manufacture	1.95	—	0.1	3.2	0.3	0.4	3.0	0.3	0.6	3.5	0.6	0.6	3.5	0.9	0.4
	10 secs.			40 secs.			1 min.			2 mins.			3 mins.		
Black ink, German ...	1.1	1.5	—	1.6	2.0	0.1	1.7	2.1	0.3	1.9	2.6	0.2	2.6	3.0	0.8
	20 secs.			1 min.			90 secs.			2 mins.			3 mins.		
Black ink, English ...	0.8	1.2	0.2	3.0	2.0	2.0	4.0	3.2	2.7	4.2	3.2	2.7	4.5	3.2	2.7

SEQUENCE OF STROKES AS PROOF OF PRIOR WRITING.

If two pen-strokes in ink cross each other while wet, the point of intersection is indicated by a heavier deposit of pigment, but it is rarely possible to ascertain which stroke is uppermost. If, however, the first stroke is dry before the second is applied, the lines are generally quite distinct, and it is usually possible, with the aid of the microscope, to decide which stroke is uppermost, or in other words more recent.

Osborn has cited some striking instances of the kind in his book, "Questioned Documents," pp. 375-393, showing that the method may give conclusive evidence of later additions to a document.

In this case of *Rex v. Cohen*, one of the alleged recent entries touched the entry immediately below it at two points, and in each case the line came below and not above the intersecting line of the following entry. Hence, there could be no doubt but that this suspected entry was written prior to those which immediately succeeded it. A conviction could not be upheld in the face of such facts as these, and the sentence on the defendant was quashed.

COPYING METHODS.

The use of the ordinary copying process for determining whether the whole of the ink upon a document is contemporaneous was suggested by Sittl (*Chem. Zeit.*, 1891, 15, 1833), who found that a good copying ink would yield a fair copy on "flimsy" paper for about eight days after writing, whilst even after four weeks a faint copy might be expected.

This method was studied by Habermann and Oesterreicher (*Zeitsch. anal. Chem.*, 1901, 40, 725), who found that under certain conditions distinct copies could be obtained by the use of a press from the moistened writing in ordinary iron-gall ink for about five months, and in exceptional cases up to nineteen months.

Carré (*Comptes rend.*, 78, 1213) had previously suggested the use of dilute hydrochloric acid (1:11) in place of water as a copying medium. He found that writing of eight to ten years of age would yield a good copy, whereas writing thirty years old have a faint unreadable copy, and ink of 1787 only yielded traces of pigment to the copying paper.

My experiments with these methods show that the results should only be accepted with great caution. If, however, there is a very pronounced difference between the copying capacity of two parts of the same document, and chemical tests indicate that the inks are of the same type and the writing is of equal intensity, it is extremely probable that the writing was not done at the same time. More definite conclusions, however, may be drawn if the ink is supposed to be an ancient one and yet gives a sharp copy.

CHEMICAL TESTS FOR AGE.

Iron-gall ink, when recently applied to the paper, reacts rapidly with various reagents, but after the lapse of a year or so will only react slowly; and, finally, after a period of six to ten years will remain unaffected for a long time. This difference was first noted by Sonnenschein (*Lehrbuch der gerichtlichen Chemie*, 1881, p. 364), and has also been recommended by Irvine (*Workshop Receipts*, Spon, fifth series, 1892, p. 88).

Various reagents have been suggested for the purpose, such as dilute hydrochloric acid, bromine water, hypochlorous acid, hydrogen peroxide, etc.

In my experience a 5 per cent. solution of oxalic acid is the most useful reagent, a drop being applied to the place on the writing by means of a capillary pipette. The effect is then followed by means of a lens, and the amount of the diffusion, if any, of blue pigment noted. It is remarkable that in old blue-black ink in writing the aniline blue, if still present, is prevented from diffusing by the insoluble tannate which has been formed. For example, in the case of the iron-gall inks of 1906, mentioned above, the specimens to which blue dye had been added did not react any more readily than the same inks without the dyestuff.

In forming any estimate of the age of ink in writing from the speed of the reaction and amount of diffusion it is essential that the dates of the inks should not be too close to each other, that the inks should be of the same type, and that the differences should be very pronounced.

All these conditions were present in the case of *Rex v. Pilcher*, which was tried in 1910. Colonel Pilcher was accused of forging his cousin's will, the alleged date of which was 1898. All the inks on the will, including the signatures of the supposed witnesses, reacted immediately with various reagents, and showed pronounced diffusion of blue pigment, the ink running all over the paper. On the other hand, a series of parallel tests applied to cheques which had been written by the alleged testatrix gave the following results:

- (1) 1907. Rapid diffusion.
- (2) 1906. Diffusion after a time, action less rapid.
- (3) 1905. July. Heavy writing. Diffusion more rapid than (2), less rapid than (1).
- (4) 1905. January. Diffusion less rapid than (3).
- (5) 1904. July. Slight diffusion, less rapid than (4).
- (6) 1904. January. Heavy writing. Diffusion fairly rapid. More pronounced than (5).
- (7) 1903. June. Very slight diffusion.
- (8) 1903. January. Very heavy writing. Diffusion very slight.
- (9) 1903. March. Medium writing. No diffusion.
- (10) 1902. June. Heavy writing. No diffusion.
- (11) 1902. February. Very heavy writing. Slight indication of diffusion.
- (12) 1902. January. Heavy writing. Very slight diffusion.
- (13) 1901. June. Heavy writing. No sign of diffusion.
- (14) 1901. February. Very heavy writing. No sign of diffusion.

Other tests proved that these were all written in the same kind of blue-black ink and of the same type as the inks on the will.

Similar results were also obtained in tests applied to a recipe book and various letters of the testatrix. I therefore concluded that the alleged will was probably not more than four or five years old at most. Eventually Colonel Pilcher confessed that he had altered the will, knowing it to be a forgery, but that he had no knowledge as to how it had been forged.

In another criminal case (*Rex v. Seddon*) a will written in ink showed no indications of having been written at a date later than was stated upon it.

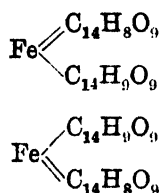
CHEMICAL CHANGES IN THE DRYING OF INK.

Hitherto the greater resistance offered to reagents by older inks on paper has been accepted as an empirical fact, and no attempt seems to have been made to give a chemical explanation of it. For several years I have made systematic experiments on this subject, and have obtained results which appear to account for the phenomena.

When solutions of ferrous sulphate and gallotannic acid are mixed, a pale ferrous tannate appears to be formed. This is indicated by the fact that any ferric salt present is immediately reduced to the ferrous condition, as is easily demonstrated by mixing ethereal solutions of ferric chloride and gallotannic acid, when an insoluble tannate is precipitated and the ether becomes bright green. But the gallotannic acid is not oxidised to gallic acid, at all events in an aqueous solution, for it can still be completely absorbed by hide powder. When the ferrous tannate solution is exposed to the air it is slowly oxidised, and forms incrustations and insoluble deposits of iron tannates. I have repeatedly separated these tannates as they formed, dried them at 100° C., and estimated the amount of iron. The results in the following table are typical of those obtained:

Iron Salts.	Gallotannic Acid, etc.	Iron in Precipitates (per Cent.).			
		1.	2.	3.	4.
Grms.	Grms.				
Ferrous sulphate :					
3	2	5.75	5.94	—	—
3	3	5.49	6.04	7.17	—
3	1	5.38	7.74	7.72	—
—	+ pine oil	8.15	—	—	—
—	+ small amount H_2O_2	14.18	—	—	—
3	2 + excess H_2O_2	24.29	21.05	22.4	21.46
3	2 + pine oil	8.15	—	—	—
—	+ saliva	7.98	—	—	—
Ferrous ammonium sulphate :					
2	2	5.08	6.90	—	—
3	2	5.36	6.16	—	—
Ferric sulphate :					
2	2	6.16	17.1	—	—
4	3	15.70	7.7	—	—
		(from filtrate diluted)			
	—	8.56	—	—	—
Ferric chloride					
Ferrous sulphate :	Galls, 2.5 per cent. extracts :				
1	Japanese galls	7.91	7.71	8.12	—
1	Aleppo galls	6.03	7.72	7.68	—
3	Chinese galls	6.86	7.56	7.57	—
3	Valonia	8.07	8.80	—	—
3	Myrobalans	5.74	—	—	—
3	Divi-divi	6.77	7.77	—	—
3	Chestnut extract	7.37	—	—	—

It will be seen that the earliest precipitates contain Fe approximating in composition to Wittstein's iron tannate, $Fe_2(C_{14}H_9O_9)_6$, containing 5.53 per cent. of iron (*Jahresber. der Chem.*, 1848, 28, 221). As the oxidation proceeds the successive precipitates contain more iron, and eventually approximate in composition to the precipitate which Pelouze obtained by exposing a solution of ferric sulphate and gallotannic acid to air for a month (*Ann. de Chim.*, 1833, 54, 337).



This compound, however, is not formed at first from ferric sulphate, but the presence of ferric salts promotes the oxidation to the more highly oxidised compounds.

I attempted to accelerate the oxidising process by adding hydrogen peroxide to the ferrous tannate. The addition of a small amount gave a precipitate containing 14.18 per cent. of Fe, approximating in composition to Wittstein's tannate with 14.21 per cent. of iron, whilst if large amounts of hydrogen peroxide were added, a heavy precipitate containing 21 to 24 per cent. of iron was immediately thrown down. This approximates in composition to Ruoss's basic tannate $(C_{14}H_7O_9)(FeO)_2$.

with 24.42 per cent. of iron (*Zeitsch. anal. Chem.*, 1902, **41**, 732). Neither of these compounds, however, appears to be formed in the normal oxidation of ferrous tannate, although if ferric sulphate is used instead of ferrous sulphate precipitates containing upwards of 16 per cent. of Fe are eventually obtained.

I then tried the effect of a catalytic agent, and added a few drops of pine oil to a solution of ferrous tannate. Immediately a dense precipitate containing 8.15 per cent. of iron was obtained. In a duplicate experiment the amount of iron present was 8.10 per cent. Hence, under these conditions, Pelouze's tannate is formed at once. In like manner, the addition of 5 c.c. of saliva accelerated the oxidation, and after two days a heavy precipitate containing 7.98 per cent. of Fe was formed. The oxidation to the more insoluble form of precipitate was thus accelerated, and it would seem that the precipitates containing amounts of iron between 5.53 and 8.0 per cent. are probably mixtures of these two tannates. Incidentally these experiments suggest a chemical explanation of the use of ink as a reagent for the development of secret writing in saliva or scent. The presence of certain substances in different kinds of galls appears to have a similar accelerating influence on the oxidation. These two tannates, when dried at 100° C., differ in properties. The first feels like soft chalk, is somewhat soluble in water, and readily soluble in dilute hydrochloric acid. The other is more granular and has a resinous appearance, and is very difficult to dissolve completely, even in strong boiling hydrochloric acid. The intermediate precipitates show increasing insolubility as the oxidation proceeds.

Here, then, we have the probable explanation of the behaviour of inks in writing on paper. The tannate first formed is sufficiently soluble to allow copies to be taken for a short time, and is readily soluble in dilute acids, but as the oxidation proceeds the resinous tannate is slowly formed, until eventually the ink becomes not only difficult to dissolve in weak acids, but also protects the soluble aniline dyestuff from the action of the reagent. When the oxidation has reached this stage, the addition of acid will slowly cause the ink to turn blue if the blue dye has not yet faded, but there will be little, if any, sign of diffusion or smudging.

DISCUSSION.

Mr. G. R. THOMPSON said he was not quite clear about one slide which showed an ink of 1906 and one of 1895. He said that from tintometric analysis of these two inks he found that they were practically identical.

				1895.	1906.
Blue	...	...	...	2.4	2.5
Red	...	...	...	1.4	1.4
Yellow	...	...	...	1.1	1.0

This is a difference of eleven years, and yet the results are similar. He asked if any light could be thrown on the age of inks by the use of panchromatic plates through screens of varying intensities. He thought it might be stronger proof if demonstration of the actual difference in colour were shown rather than a string of figures of tintometric results.

With regard to the estimation of iron, was this done on the actual inks or from the writing on the documents?

Mr. BUTTERFIELD inquired to what extent concentration and ageing or oxidation of ink which had been left standing in the inkpot would affect the judgment as to the age of the writing. He said he sometimes used a fountain pen filled with copying ink, but if the pen ran out he dipped it into an inkpot which was rarely used, and might have ink filled from the same stock bottle as the fountain pen, but which had been exposed for three or four months in the loosely-covered inkpot. He noticed that the writing with the fountain pen was totally different from that written with the stale ink from the inkpot. He wondered to what extent judgment as to the age of two consecutive lines of writing of the same letter would be vitiated by such a change from fresh to stale ink of the same original supply, which would occur merely from the accident of a fountain pen running dry between the two lines.

Mr. CHASTON CHAPMAN said that most of the ink supplied to-day, even by the best makers, appeared to throw a considerable deposit in the inkpot after a few weeks, and inquired whether the age of the ink at the time of writing might not seriously affect the conclusions drawn from an examination of the writing at some subsequent date.

He (Mr. Chapman) had also been wondering what would be the effect of writing with a pen which had just been moistened with saliva. If, for example, a person were to wet a new nib with saliva before writing his signature, and another person were a little later to dip the pen into the same ink and append his signature to the same document, would there be any difference between the two writings such as might lead to the conclusion that the same ink had not been used in both cases?

He would also like to ask Mr. Mitchell whether, in the case of writing with inks containing mixed dyes on common and slightly absorbent paper, he had ever observed any tendency in the direction of the separation of the dyes, owing to the capillary action of the fibres. If so, such separation might materially affect the mixed colour value as determined by the tintometer.

In reply to Mr. Thompson's questions, Mr. MITCHELL said that the determination of the iron was made on the inks themselves specially prepared. There was no doubt that old ink in writing resisted reagents much more than new inks, and it was a reasonable hypothesis to draw conclusions from the behaviour of the deposits thrown down by inks. In some cases attempts had been made to follow the action with a stop-watch, but conclusions from such data were dangerous. When a crust formed on the ink, it was in all probability very similar to what was formed when the ink was placed on the paper.

With regard to the effect of oxidation of the ink in the bottle, Mr. Mitchell said that in the case of insufficiently matured ink there was generally a deposit, but the deposit thrown out was, as a rule, not iron tannate, but some form of tannin. Even if the ink had become oxidised in the pot, the oxidation would continue on the paper. In the case of copying inks the problem was much more complicated. There would be a very decided difference in the behaviour of two inks known to be copying inks, for it was dangerous to compare copying ink with ordinary ink. The change by oxidation was very much slower in the case of copying ink than with ink without the extra amount of gum and pigment which copying inks contained.

In reply to Mr. Chapman's question, Mr. Mitchell said that the only distinction would be a chronological one. The ink of 1895 would not react so readily as one of 1906, but it would not be possible to state that one ink belonged to one period or the second to the other. As a test of the age of writing the tintometer was only useful for comparing fairly recent inks on paper.

With reference to Mr. Thompson's suggestion of the use of colour screens, Mr. Mitchell remarked that he had tried the use of screens and colour sensitive plates some years ago, and found that the main difference in the results was due to differences in intensity rather than in the colour of the inks.

In reply to Mr. Chapman's suggestion with regard to the effect of absorbent paper in separating dyes, Mr. Mitchell said that this method had already been used for separating the pigments in inks, but it would not be practicable in the case of dried ink on paper.

The action of saliva on the inks in writing required further investigation, but presumably wetting a new pen would only affect one or two letters and would be more likely to cause a deposit on the inkpot.

With regard to carbon ink, as a rule it could easily be washed off the paper, since carbon ink did not penetrate into the fibres. The nearest approach to a result of that sort was obtained with a fine suspension of colloidal graphite, which was not at all a bad carbon ink when mixed with a certain amount of water, as the particles did not separate out easily.

With regard to the question of dye in the ink in writing and the blue colour being reproduced on the addition of an acid, Mr. Mitchell said that it was quite possible in cases where the dye did not run that the fixation of the dyestuff might be due not merely to its being mechanically retained by an enveloping resinous tannate, but also to chemical combination.



A RAPID VOLUMETRIC METHOD FOR THE ESTIMATION OF IRON. APPLICABLE IN THE PRESENCE OF HYDROCHLORIC ACID, PHOSPHORUS, OXY-ACIDS, AND ORGANIC MATTER.

By H. DROOP RICHMOND, F.I.C., AND EDITH M. ISON, B.Sc., A.I.C.

(*Read at the Meeting, May 5, 1920.*)

Owing to the presence of sugar, phosphorus compounds, and other compounds in syrups, the conventional methods of iron determination are not applicable, and as much of the therapeutic effect of the syrups is due to the iron present, it is highly desirable to be able to estimate its amount rapidly and with accuracy.

The method we describe, which is both rapid and accurate, depends on the following facts:

1. In 5N hydrochloric acid solution iron is completely ionised, even if phosphates and organic matter be present.

2. Permanganates oxidise ferrous ions to ferric ions in the presence of sugars, and also destroy the colouring matters used in the syrups. In oxidising ferrous to ferric ions by standard permanganate, the amount used before the colour is discharged shows that oxidation of the ferrous ion takes preference over that of the dye, while the sugar is attacked in a still less degree.

3. A small excess of permanganate is reduced completely by the sugars, and practically no chlorine is liberated.

4. Ferricyanides are sensitive indicators for the production of ferrous ions, and thiocyanates for their removal.

5. Stannous chloride reduces ferric to ferrous ions, and in an atmosphere of carbon dioxide the reduction is a measure of the iron present.

We operate thus: 5 or 10 c.c. of the syrup is measured (or weighed) into a 100 c.c. flask, a few drops of strong hydrochloric acid added, and $\frac{N}{10}$ permanganate solution run in till a transitory purple is produced throughout the solution, the colour of such syrups as *Syrupus ferri phosphatis compositus* or *Syrupus ferri glycerophosphatis compositus* being destroyed during the addition of the permanganate. The purple colour vanishes rapidly, and usually the solution contains no free chlorine, and the iron present is entirely in the ferric state. An equal bulk of strong hydrochloric acid is added, and a little sodium bicarbonate dropped in to give an atmosphere of carbon dioxide in the flask; 1 drop of $\frac{N}{10}$ stannous chloride solution is added, and a drop of the mixture added to a drop of a freshly prepared ferricyanide solution on a white tile; a blue coloration generally results, except in cases where a great excess of permanganate was added, and in these cases stannous chloride must be added till a reaction for ferrous iron is given. The solution, which has the usual bright yellow colour of ferric salts in strong hydrochloric acid, is now titrated till the colour becomes faint, and the titration continued till a drop no longer colours a drop of a solution of a thiocyanate on a white tile. The number of c.c. used multiplied by 0.0056 will give the weight of iron present.

With careful oxidation the use of the ferricyanide indicator may be dispensed with, and with non-coloured solutions the disappearance of the yellow marks the end point.

The results below show the accuracy of the method:

Iron solution containing by gravimetric analysis 0.837 grm. Fe per 100 c.c.; aqueous solution, 0.833; after re-oxidising, 0.832; sugar solution, 0.835.

Syrupus ferri phosphatis, B.P. theory, 0.87 per cent. (three samples): 0.835 and 0.825, 0.86, 0.868.

Syrupus ferri phosphatis compositus theory, 0.437 per cent.: 0.437 and 0.422.

Another sample + 5 per cent. extra iron, theory 0.46 per cent.: 0.46.

Syrupus ferri phosphatis compositus, another sample strong in iron: 0.482 and 0.485.

Syrupus ferri glycerophosphatis compositus, B.P.C. theory: 0.087 per cent. (two samples): 0.088 and 0.087, 0.084.

Syrupus ferri hypophosphatis, made to contain 0.48 per cent.: 0.46, 0.49, and 0.48.

Easton's syrup theory, 0.87 (three samples): 0.835, 0.88, 0.85.

Ferri phosphas saccharatus (one of strength below B.P. requirements, one of B.P. strength) : 51.8, 64.1 ; Romijn's method, 51.4, 64.6.

Syrupus glycerophosphatis cum formatibus, B.P.C. theory, 0.087 per cent. (known to be low) : 0.069, 0.067 ; after oxidation, 0.066.

Glycerinum glycerophosphatum compositum, B.P.C. theory : 0.081, 0.087.

We find that if the stannous chloride solution be kept in an atmosphere of hydrogen, it loses strength at so slow a rate that occasional checking of its titre only is required. In the absence of organic matter this method may be combined with a permanganate titration in hydrochloric acid solution thus : the iron having been brought to the ferrous state by any of the usual methods, the solution is titrated with $\frac{N}{10}$ permanganate ; half the volume of strong hydrochloric acid is added, and the solution titrated back with $\frac{N}{10}$ stannous chloride, using ferricyanide as indicator, and the amount used subtracted from the permanganate titration, and the remainder gives an estimation of the iron. The titration with stannous chloride may be continued to the thiocyanate end point, and gives another iron estimation which agrees closely with the corrected permanganate figure.

This work was carried out in the analytical laboratory of Boots' Pure Drug Company, Ltd., to whom our thanks are due.



THE ESTIMATION OF NITROGLYCERINE.

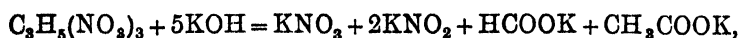
By H. DROOP RICHMOND, F.I.C.

(Read at the Meeting, June 2, 1920.)

NITROGLYCERINE is used in medicine, a 1 per cent. solution being official in the B.P. under the name of *Liquor trinitrini*, and in the U.S.P. as *Spiritus glycerylis nitratis*. The test given in the B.P. is a dilution test with water, and useless as a quantitative indication ; the U.S.P. prescribes the weighing of the residue after spontaneous evaporation and drying over sulphuric acid.

In the hope of finding a simple routine method for estimating nitroglycerine, not only in the official preparations, but in the stronger solutions marketed under the name of *Liquor glonoini*, I have examined many of the methods proposed.

Hay (*Trans. Royal Society of Edinburgh*, 32, 67 ; also *Chem. Centralb.*, 1884, 108) concluded that the action of alkali is—



and suggests the estimation of the nitrite. Allen ("Commercial Organic Analysis, third edition, vol. ii., part i., 335) states that he has attempted to measure the potash required for saponification, and though finding fairly concordant results, mentions that the dark colour of the solution prevents the end point being distinct ; as he gives no figures, it is to be presumed that he did not consider this a practicable method. Hempel

(*Zeitsch. anal. Chem.*, 1881, 82) dissolves the nitroglycerine in sulphuric acid, and measures the nitrous oxide given off in a nitrometer; but states that 5 c.c. only of sulphuric acid must be used, otherwise the results are low. He does not find saponification to give good results.

Champion and Pellet (*Ann. Chim. anal. Appl.*, 1900, 5, 365) have devised methods depending on the reduction of the nitro groups to nitrous oxide by ferrous sulphate. The Kjeldahl-Jodlbauer method for estimating nitrogen and reduction by stannous and titanous chlorides are used for the estimation of nitro-nitrogen.

Siberrad, Ablett, and Merryman (*J. Soc. Chem. Ind.*, 1906, 25, 628) heat for six hours with sodium ethylate, and reduce the nitrate and nitrite formed to ammonia with a suitable alloy (*cf.* Lowry, Browning and Farmery, *J. Chem. Soc.*, 1920, 117, 552).

My first experiments were the evaporation of the alcoholic solution at a low temperature, solution of the residue in sulphuric acid (5 c.c.), and estimation of the nitrous oxide given off by shaking with mercury in a nitrometer; but as I obtained 89.0 and 65.1 per cent. of the theoretical quantity in two experiments, I did not pursue the method further.

Next I attempted to evaporate the solution after adding sodium hydroxide, dissolving in small amounts of water, in which the residue was very soluble, and making a nitrometer estimation. The results were :

					Per Cent. of Theory.
Approx. 8 molecules NaOH,	rapid	evaporation	...		105
"	"	slow	"	...	132, 107
Approx. 4	"	rapid	"	...	97.5, 96.0, 96.0, 99.5
"	"	slow	"	...	101

Although fairly concordant results were obtained by rapid evaporation in the presence of limited amounts of alkali, the method was considered to be dependent too much on conditions difficult of attainment.

Attempts were made to estimate the nitrous oxide given off by treating the residue after evaporation with alkali with potassium iodide and sulphuric acid in a nitrometer. The results were :

					Per Cent. of Theory for per Cent. - NO ₂ .
Approx. 8 molecules NaOH,	rapid	evaporation	...		102.6, 90.4, 104.4, 119.6
" 4	"	"	"	...	95.7, 87.9, 89.7

In two cases the residue after evaporation was made slightly acid, and air passed over the solution till all smell of nitrous fumes was removed, and the nitrate estimated in the nitrometer; 94.8 and 114.6 per cent. of theory for 1 - NO₃ were given in two experiments. An experiment was made by adding eight molecules NaOH to the alcoholic solution, and allowing the mixture to stand for twenty hours, decanting the alcoholic portion, and washing the brown syrupy residue with alcohol. The alcoholic solution was evaporated, and gave total nitrogen, 103.8 per cent. of theory; nitrous nitrogen, 98.2 per cent.; nitric nitrogen, 94.2 per cent.; while the syrupy residue gave no nitrous oxide in the nitrometer, but an appreciable amount of CO₂.

An attempt was made to estimate the nitrous nitrogen in the residues after evaporation with 8NaOH by the urea method.

The figures were :

	Total Gas at N.T.P. from 0.05 Grm.	Of which N ₂ .
Short evaporation ...	31.4 c.c.	Not estimated.
Medium evaporation ...	35.7 c.c.	17.5 = 89.3 per cent.
Long evaporation ...	40.7 c.c.	17.85 = 91.1 per cent.

Some experiments were tried by adding 8NaOH, allowing the solution to stand in the cold, pouring the alcoholic solution direct into the nitrometer, adding potassium iodide solution and dilute sulphuric acid, as in the B.P. method for examining *Spiritus atheris nitrosi*, and measuring the gas. The results were :

Time of Standing.	Per Cent. of Theory.	Per Cent. of 95.7.
5 minutes... ..	91.5	—
30 "	96.3	100.6
30 "	94.6	98.9
1 hour	95.4	99.7
3 hours	96.8	101.1
4 "	95.4	99.7

Or a mean of 95.7, excluding the five-minute results.

This appeared to be a rough empirical method, giving results of sufficient accuracy for control purposes.

The details are : to 5 c.c. of *Liquor trinitrini* in a corked test-tube add 0.2 c.c. 7.5N NaOH solution; after one-half to two hours pour into the cup of a nitrometer filled with saturated brine, rinse the tube with small amounts of alcohol, and add 5 c.c. of a with 10 per cent. solution of potassium iodide and then 5 c.c. 3N sulphuric acid. Shake till evolution ceases, measure the gas, and correct to N.T.P. number of c.c. at N.T.P. $\times 0.1062$ = per cent. nitroglycerine w/v.

Estimations made by treating the alcoholic solution with 8NaOH for five minutes, one hour, and eighteen hours, and estimating the nitrogen by Devarda's method, gave 90.0, 92.3, and 88.6 per cent. of theory, but if a small quantity of hydrogen peroxide were added before the alkali, practically theoretical results were obtained. It is evident from the above results that Hay's equation does not represent the change that takes place; CO₂ was detected in the gas in the nitrometer on several occasions, though it was proved that during the evaporation of alcohol with the amount of caustic soda used not enough CO₂ was absorbed from the atmosphere to give any in the nitrometer. It was noticed on adding the alkali that the alcoholic solutions almost immediately became turbid, that heat was evolved as noted by Hay, and that a dark syrupy layer was deposited on the bottom of the vessel in a short time; the alcoholic solutions were not very dark.

Notwithstanding Hempel's criticism, estimations were made of the alkali required for hydrolysis; in the first series 25 c.c. $\frac{N}{2}$ alcoholic soda solution were added to 10 c.c. of a 5 per cent. solution in 90 per cent. alcohol, allowed to stand for various times, and the excess titrated with $\frac{N}{2}$ acid without disturbing the syrupy layer, with results as follows :

Time of Standing.	c.c. $\frac{N}{2}$ Alkali used per Grm.
22 hours	19.50
22 "	19.42
1 hour	18.77
15 minutes	18.18
5 "	17.79
2 "	17.72

It was seen that if the results were plotted out as a curve, the point for zero time would be very close to 17.62, the theoretical amount for 4NaOH ; it was thought that the higher results were probably due to the retention of some alkali in the syrupy phase as well as possibly to the gradual formation of carbonates. It was found that the one-hour titration, when violently shaken, became alkaline again, and required 0.6 c.c. $\frac{N}{1}$ acid per grm. to become neutral, although all the syrup could not be removed from the bottom by shaking.

The deposit in one of the twenty-two-hour titrations on solution in water required 1.45 c.c. per grm.

It appeared probable that no further alkali was being absorbed in any change that was taking place, but was simply being removed from solution. Another series was started in which the alkali was allowed to act for a limited period, and the solution shaken violently during titration. A solution of soda in methylated spirit, one of soda in pure alcohol, and one of potash in pure alcohol, all nearly $\frac{N}{2}$, were used but the only difference observed was that the potash caused a slightly greater darkening than the soda in all cases; when a few c.c. of alkali had been added, the solution became turbid and slightly warm, and evidently the action was very rapid.

Ten c.c. of 5 per cent. solution and 25 c.c. of approximately $\frac{N}{2}$ alkali were used, and titrated back with $\frac{N}{2}$ acid. The results were:

				c.c. N per Grm.
1.	KOH, 2 minutes	...	...	17.28, 17.18, 17.17
	KOH, 5 "	...	...	17.22, 17.28, 17.21
	NaOH, 2 "	...	...	17.28
	NaOH, 5 "	...	...	17.28, 17.19, 17.23

Or a mean of 17.23, which is equivalent to 3.909 molecules. This is equal to 97.8 ± 0.3 of the theoretical quantity, assuming that four molecules are required.

The concordant results were found, however, to be due to the same conditions being observed, for in the next series the conditions were the same, except that $\frac{N}{1}$ acid was substituted for $\frac{N}{2}$, thereby introducing less water, with results as follows:

2. All five minutes: 17.64, 17.58, 17.78, 17.80, 17.80, or a mean of 17.72 c.c. per grm.

3. Two c.c. of a 5 per cent. solution + 8 c.c. alcohol with 10 c.c. $\frac{N}{2}$ alcoholic soda gave, titrated with $\frac{N}{2}$ acid, 16.25, 15.70, 15.95, a mean of 15.95 c.c. per grm.

4. Four c.c. of a 5 per cent. solution + 16 c.c. alcohol with 10 c.c. $\frac{N}{2}$ alcoholic soda gave, titrated with $\frac{N}{2}$ acid, 17.90, 17.88, 17.42, 17.80, a mean of 17.75 per grm.

5. Two c.c. of a 5 per cent. solution + 8 c.c. alcohol with 10 c.c. $\frac{N}{2}$ alcoholic soda gave, titrated with $\frac{N}{2}$ acid, 17.12 c.c. per grm.

6. Ten c.c. of 5 per cent. solution with 25 c.c. $\frac{N}{2}$ alcoholic solution, titrated back with $\frac{N}{1}$ acid in 80 per cent. alcohol, gave 17.64, 17.64, 17.54 c.c. per grm.

It is evident that the final alcoholic strength influences the result; tabulating alcoholic strength and c.c. per grm. we get:

Final Alcoholic Strength by Volume.		c.c. per Grm.
3	66	15.95
1	75	17.23
5	77	17.12
2	81	17.72
4	81	17.75
6	90	17.60

The experiments show that four molecules of alkali are necessary, and—

1. Potash and soda give the same results.
2. Variation of the concentration of nitroglycerine from 0.5 grm. in 35 c.c. to 0.2 grm. in 30 c.c. produced no appreciable variation.
3. Excess of alkali varying from 43 per cent. to 200 per cent. did not make differences outside the limits of experimental error; it was found, however, that when the excess was dropped to 27 per cent. the results were low (16.70 c.c. per grm.).
4. A final alcoholic strength of 81 per cent. was necessary.

The acidity of an alcoholic solution of nitroglycerine could be titrated by adding to 10 c.c. 50 c.c. of water and titrating with baryta, taking as the end point the appearance of a pink colour throughout the whole solution, permanent for a second or two; this end point, though fugitive, was sufficiently definite. Results are :

Acid added as c.c. $\frac{N}{10}$.	c.c. $\frac{N}{10}$ used.	Acidity calculated.
X	Y	
0.0	0.1	0.0
1.75	1.95	1.8
2.0	2.25	2.1
2.5	2.65	2.5
5.1	5.3	5.1
12.5	12.65	12.3

There was evidently a slight hydrolysis, and the results were calculated by the formula $X = \frac{Y - 0.1}{1.02}$

The agreement is satisfactory, and shows that acidity can be estimated with sufficient accuracy.

The method for the analysis of the commercial nitroglycerine solution is : to 5 c.c. of a 10 per cent. solution + 5 c.c. 90 per cent. alcohol, or 10 c.c. of a 5 per cent. solution, add 25 c.c. $\frac{N}{2}$ alcoholic sodium hydroxide, or to 20 c.c. of a 1 per cent. solution add 10 c.c. ; allow the mixed solutions to stand for about five minutes, add phenolphthalein, and titrate back with $\frac{N}{2}$ acid, shaking violently when near the end point, which is quite sharp. To avoid adding too much water, alcoholic $\frac{N}{2}$ acid may be used for titration of the excess of alkali.

If more than 3 to 4 c.c. of acid are required, the determination must be repeated, increasing the alcoholic strength, as the amount of water introduced must not exceed 10 per cent. of the total volume. Correct the volume of alkali absorbed, expressed as c.c. $\frac{N}{2}$ for the acidity, estimated as above, and the weight of nitroglycerine will be the c.c. of $\frac{N}{2} \times 0.05675$. The results above in eleven experiments gave from 98.9 to 101.5, mean 100.4.

By adding 2 c.c. of 100 vol. hydrogen peroxide before the addition of alkali no discoloration takes place, and a white precipitate settles; the results correspond closely to the absorption of 4 molecules, and the final alcoholic strength has much less influence on the titration. I recommend this modification.

The sp. gr. of nitroglycerine is 1.600, and that of 90 per cent. (by vol.) alcohol is 0.8337, and if made up strictly according to the B.P. directions the sp. gr. of *Liquor*

trinitrini should be 0.8385, and not 0.840, as stated in the B.P.; there is no appreciable alteration in volume on dissolving nitroglycerine in alcohol; and the sp. gr. of a solution of 50 grms. in 1 litre being found to be 0.8576, the figure calculated assuming no contraction.

It is evident that the equation given by Hay does not express the reaction which takes place; the deposition of the syrupy precipitate, the formation of carbonates in quite large amounts, the presence of aldehyde and ammonia (both of which were detected), as well as the fact that only four molecules of alkali are required, all show that an action of a different nature occurs. By the action of two molecules or less of alkali I have found that the amount of nitrite formed is practically equivalent to the alkali added, and I suggest that the first action is the formation of two molecules of alkali nitrite and a molecule of mono-nitroglycerine peroxide; from this, one molecule of alkali nitrate and one of an alkali glycerate is produced by the further action of alkali. The alkali glycerate partly splits up into aldehyde and alkali carbonate, and the aldehyde reduces some of the nitrite to ammonia and possibly other nitrogen compounds; some of the nitrogen would be lost as nitrogen gas, owing to the formation of ammonium nitrite. In the presence of hydrogen peroxide the peroxide is catalytically decomposed with the formation of a hydroxypyruvate. Acetic acid found by Hay would result from the oxidation of the aldehyde.

This work was carried out in the analytical laboratories of Boots' Pure Drug Company, Ltd., to whom my thanks are due.

DISCUSSION.

Mr. DUPRÉ remarked that there are few explosives which can be treated with alkali in which nitroglycerine would be the only body present which reacts with it even after extraction. He said it was a most difficult question; there are many reactions like this one, where small subsidiary reactions prevent one getting a theoretical result. For instance, oxidation by permanganate and estimation of oxalic acid found will sometimes give good results, but unless the estimations are carried out under exactly the same conditions of temperature and concentration, consistent results cannot be obtained. He said that a point which required elucidation was the estimation of nitroglycerine in the presence of partially nitrated bodies, such as mono-nitro- and tri-nitro-toluol. This could not be done satisfactorily by means of the nitrometer, because the nitro-bodies become further nitrated, and the resulting compounds not being acted upon by the acid and mercury, a loss of nitric oxide occurs.

One method which is fairly satisfactory is to make up a mixture of nitroglycerine and nitro-compounds containing the proportions of nitro-compounds probably present in the explosive under examination, make the estimation in the nitrometer, and from the results obtain a correction to be applied to the figure given by the explosive itself.

Much the same trouble would occur if alkali were used; all these nitro-bodies inter-react with alkali, and again incorrect results are obtained. A method is needed in which a definite reaction with nitroglycerine is obtained and which is not affected by nitro-bodies.

Mr. G. NEVILL HUNTLY asked if Mr. Richmond had tried a reduction method, and suggested the use of titanous salts for this purpose.

Mr. BROWNING said that in one stage of the reaction (cordite, lime, and pyridine water) nitric oxide is liberated, even when a rapid current of air is passing through it. The quantity is, however, much reduced when air is present.

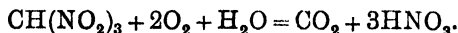
Mr. RICHMOND, in reply, said that with regard to the question of titanous chloride and stannous chloride they were both suitable, but gave results which were rather low, the explanation of which was shown in a paper in a recent number of the *Journal of the Society of Chemical Industry* by Callan, Henderson, and Strafford, in which it was proved that chloramines are apt to be formed, and titanous sulphate was recommended, which gave practically theoretical results. He had not observed the evolution of nitric oxide, but the quantities used did not exceed 0.5 gm., and it might easily have been missed in small amount. With reference to Mr. Dupré's remarks, his criticism was quite just, and he (Mr. Richmond) had seen such difficulties, and for that reason confined his paper to the pharmaceutical estimation, but possibly the extreme rapidity of the hydrolysis of nitroglycerine might serve as a basis for its estimation in the presence of other substances.



ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Estimation of Nitroform by Potassium Permanganate. P. V. McKie. (*J. Chem. Soc.*, 1920, 117, 646-648.)—Nitroform may be accurately estimated in the presence of nitric, nitrous, and sulphuric acid by oxidation with potassium permanganate solution—



Twenty-five c.c. of the nitroform solution are added to a measured excess of $\frac{\text{N}}{10}$ permanganate solution acidified with 10 c.c. of 10 per cent. sulphuric acid, and the mixture heated for one minute (not more) at 30° C. (not higher); then treated with 20 c.c. of 2.5 per cent. potassium iodide solution, and the liberated iodine titrated with $\frac{\text{N}}{10}$ thiosulphate solution. Or any nitrous acid may be destroyed by boiling the liquid for about a minute with ammonium nitrate (3 grms. per 0.2 to 0.3 gm. of nitrous acid) prior to the oxidation at 30° C. Or the nitroform may be distilled in a current of steam (100 c.c. acidified with 15 c.c. of phosphoric acid, sp. gr. 1.7) after treatment with ammonium nitrate, and the distillate titrated. C. A. M.

Separation of Saccharin and Benzoic Acid. E. Schowalter. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1919, 38, 185; through *Chem. Zeit. Übersicht*, 1920, 44, 138.)—Benzoic acid and saccharin are extracted together in the usual methods for the estimation of benzoic acid, and the two substances cannot be separated from each other by sublimation, since they both begin to sublime at about 120° C. Saccharin and its sodium salt are insoluble in carbon tetrachloride, and this solvent may be

used for the separation of the benzoic acid. The ethereal solution of the two substances obtained as described in Von der Heide and Jacob's method for the detection of benzoic acid (ANALYST, 1910, 35, 166) is evaporated at a low temperature, the residue is dried under reduced pressure if it contains acetic acid, and is then extracted with carbon tetrachloride. The dissolved benzoic acid may be titrated, whilst the residue insoluble in carbon tetrachloride may be tested for the identification of the saccharin. Saccharin and benzoic acid are absorbed to a considerable extent by animal charcoal, and this should not be used for the preliminary decolourisation of coloured solutions.

W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Volumetric Estimation of Uric Acid by a Modification of Blarez and Tourrou's Method. G. Pegurier. (*Ann. Chim. anal.*, 1920, 2, 109-110.)—Fifty c.c. of the urine are mixed with 5 c.c. of saturated sodium carbonate solution, and to the mixture are added 5 c.c. of Fehling's solution previously decolourised by the careful addition of sodium bisulphite solution. The Fehling's solution used contains 130 grms. of sodium hydroxide, 105 grms. of tartaric acid, 80 grms. of potassium hydroxide, and 40 grms. of copper sulphate per litre. The precipitate formed is collected after five minutes, washed several times with water, the filter and precipitate are placed in a beaker containing 150 c.c. of water, 10 c.c. of dilute sulphuric acid (1:1) are added, and the mixture is titrated with permanganate solution. If the latter contains 2.12 grms. of potassium permanganate per litre, each c.c. is equivalent to 0.1 gm. of uric acid per litre of urine.

W. P. S.

ORGANIC ANALYSIS.

Identification of Acids. V. *p*-Halogen Phenacyl Esters. W. L. Judefind and E. E. Reid. (*J. Amer. Chem. Soc.*, 1920, 42, 1043-1055.)—Organic acids may be identified by preparing their *p*-halogen phenacyl esters, many of which have distinctive m.-pts. The *p*-halogen phenacyl bromides, and especially *p*-bromophenacyl bromide, are the most useful reagents for this purpose, particularly in the case of monobasic aliphatic acids. The method of preparation is analogous to that used in the preparation of *p*-nitro-benzyl esters (ANALYST, 1917, 42, 89), monobasic acids being heated for three-quarters to one hour with the reagents, dibasic acids for two hours, and tribasic acids for three hours. When possible, the alkali salts of the acids are used, otherwise the free acid is nearly neutralised with sodium carbonate prior to adding the reagent and alcohol. The resulting esters are recrystallised until a constant m.-pt. is obtained. The following m.-pts. were observed with the *p*-bromo-phenacyl esters of typical acids: Acetate, 85.0°; benzoate, 119.0°; butyrate, 63.0°; caprate, 66.0°; caprylate, 65.5°; citrate, 148.0°; lactate, 112.8°; palmitate, 81.5°; salicylate, 140.0°; stearate, 78.5°; succinate, 211.0°; thiocyanate, 146.5°; and valerate, 63.6° C.

C. A. M.

Estimation of Alcohol in Mixtures of Alcohol, Sulphuric Acid, and Water. G. S. Butler and H. B. Dunncliff. (*J. Soc. Chem. Ind.*, 1920, 39, 146t.)—The method described involves the estimation of the alcohol both as ethyl hydrogen

sulphate and as free alcohol. If the free sulphuric acid is estimated by titration by the described method, the results show good agreement with those obtained by the precipitation of the free acid as barium sulphate, thus showing that during titration no appreciable hydrolysis takes place. Combined alcohol is estimated by determining the amount of acid which has undergone esterification. A weighed quantity of the alcoholic sulphuric acid is titrated against standard alkali; this "direct" titration gives (b) the percentage of acid present in terms of H_2SO_4 —i.e., the free sulphuric acid and the acid due to ethyl hydrogen sulphate. The "total" acidity is found by evaporating a weighed quantity of the alcoholic sulphuric acid to a pasty consistence with excess of standard caustic soda or potash, heating in an air oven at $120^\circ C.$ for half an hour, and then, after extraction with distilled water, titrating the excess alkali with standard acid. From this is calculated the percentage acidity of the alcoholic sulphuric acid before any esterification took place. The value is expressed as percentage of sulphuric acid (a). Thus, the percentage of acid esterified = $2(a - b) \div a \times 100$. The weight of esterified alcohol in 100 grms. of the alcoholic sulphuric acid is $\frac{2(a - b)}{a \times 100} \times \frac{46}{98}$ grms. The free alcohol may be determined by weighing out 25 grms. of the alcoholic sulphuric acid, making faintly alkaline with sodium carbonate, and distilling until about 95 c.c. of distillate has been collected. This treatment does not hydrolyse the ester, and the distillate contains all the uncombined alcohol present. It is made up to 100 c.c., and its density at $60^\circ F.$ determined. Analyses are appended of mixtures of known content which were determined with exactitude by the method described.

H. F. E. H.

Butyl Alcohol as a Medium for the Determination of Saponification Numbers. A. M. Pardee, R. L. Hasche, and E. Emmet Reid. (*J. Ind. and Eng. Chem.*, 1920, 12, 481.)—Normal butyl alcohol is found by the author to be superior to ethyl alcohol as a medium for quantitative saponification of fats and waxes, since satisfactory results are more readily obtained, and special precautions less often required. Substantially the same values result with both alcohols for the more readily saponified oils and fats, although the values in butyl alcohol are always somewhat higher. For substances which are difficult to saponify, such as wool fat, lanolin, and beeswax, considerably higher and more reliable values are obtained by using butyl alcohol than in the same volume of ethyl alcohol, though the latter values would doubtless have reached the higher figures had larger amounts of ethyl alcohol been employed or had the saponification been carried on longer. A table is given showing comparative values for twenty different kinds of oils, fats, and waxes, determinations in all cases being made on 1 gm. quantities, and 10 c.c. of the standard $\frac{N}{2}$ alcoholic alkali being employed for the heating on a steam bath for exactly one hour. Fifty c.c. of carbon dioxide free water were then added, and excess alkali titrated with $\frac{N}{2}$ hydrochloric acid; since the butyl alcohol was nearly anhydrous, 0.5 c.c. of water was added to each flask containing this alcohol half an hour after heating had begun to provide for the saponification of the butyl ester which is first formed.

H. F. E. H.

Tanning Value of Vegetable Tanning Materials. J. A. Wilson and E. J. Kern. (*J. Ind. and Eng. Chem.*, 1920, 12, 465-469.)—It has become customary to apply the name tannin to that portion of the water-soluble matter of certain vegetable materials which will precipitate gelatin from solution and which will form compounds with hide fibre which are resistant to washing. The remaining portion of the soluble matter is called non-tannin. A convenient amount of the tannin material is shaken with a definite amount of purified hide powder until all tannin has been removed from solution. This point is determined by filtering off a portion of the residual liquor and adding drop by drop, avoiding a large excess, a solution containing 10 grms. of gelatin and 100 grms. of sodium chloride per litre; if the solution becomes turbid or a precipitate forms, it shows that all tannin has not been removed from solution, in which case the mixture must be discarded and the test repeated, using less of the tannin material or shaking for a longer time, until the solution after filtration gives no visible reaction with the gelatin-salt reagent. The tanned powder is then washed free from soluble matter, including the non-tannin removed from solution by the hide powder, which is responsible for the large errors in the methods now in use. It is then carefully dried and analysed for tannin as in the regular procedure for vegetable-tanned leathers, and from this figure the percentage of tannin in the original material may be readily calculated. Since the results obtained for any one material, using different dilutions, agree closely, it is concluded that the hide powder removed practically all of the tannin from all solutions which gave no test with the gelatin-salt reagent after the shaking process. In each test, 12 grms. of hide powder (of known hide substance content) were put into a wide-mouth, rubber-stoppered, half-pint bottle, the tanning material dissolved in 200 c.c. of solution was added, and the whole was shaken in a rotating box for six hours. Whenever the liquor after the six-hour shaking gave a turbidity or precipitate with the gelatin-salt reagent, the test was repeated with less material. The smallest amount used for any material represents the least it was possible to use and still get reproducible results. The tanned powder was washed by shaking with 200 c.c. of water for thirty minutes, squeezing through a suitable cloth, and repeating the washing operation until the wash water showed no colour and gave no test with ferric chloride solution. The washed powders were dried at room temperature for twenty-four hours or longer, and then analysed for water, ash, fat, and hide substance. The difference between 100 and the sum of these percentages was taken as the percentage of tannin in the leather. The parts of tannin per 100 parts of hide substance in the leather divided by parts of tanning material used per 100 parts of hide substance gave the fraction of tannin in the original material. The principle of the official (American) method is similar to that of the new method as regards the shaking of a solution of the tanning material with hide powder, but differs in that the drop in concentration of the liquor upon shaking is taken as the measure of its tannin content, while the tanned powder is simply discarded. As might be expected, the greatest errors in the official method are obtained with those materials containing the greatest proportion of non-tannin to tannin. Quebracho, having least non-tannin, gives the smallest error. The analyses of eight common tanning materials by the new method and by the official method of the American

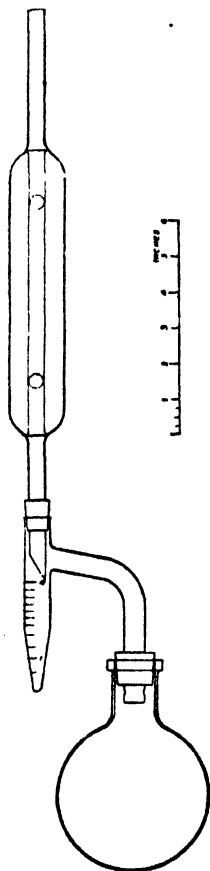
Leather-Chemists' Association indicate that the latter method is in error to the extent of from 43 to 220 per cent. H. F. E. H.

Convenient Method for the Determination of Water in Petroleum and other Organic Emulsions. E. W. Dean and D. D. Stark. (*J. Ind. and Eng. Chem.*, 1920, 12, 486-490.)—Experience has indicated that the method of

distilling in the presence of an immiscible solvent is the most generally satisfactory scheme of estimating the water content of organic emulsions, particularly petroleum emulsions. The authors have found the most satisfactory solvent to be a so-called cleaner's naphtha derived from petroleum. A mixture of 80 per cent. of commercial xylene and 20 per cent. of commercial 90 per cent. benzol has also been found satisfactory. This mixture should not be saturated with water before using. The use of a charge of 100 c.c. of emulsion and 100 c.c. of solvent has proved satisfactory for oils containing between 0.5 and 10 per cent. of water. If an accurate determination of water content of less than 0.5 per cent. is desired, it is necessary to use 200 c.c. or more of both emulsion and solvent, employing, of course, a correspondingly larger distilling flask. If the emulsion to be tested contains more than 10 per cent. of water, the quantity taken should be some suitable fraction of 100 c.c. The method has been tried with a number of emulsions prepared by mixing known amounts of water with anhydrous oils. Results have almost invariably checked the theoretical water content within ± 0.1 c.c., which apparently represents the normal magnitude of error. The applicability of the method for the separation of water from refractory emulsions has been questioned, and tests have been made to settle this point. Water was determined according to the procedure described. The flasks were allowed to cool somewhat, then connected with an ordinary distilling tube and condenser, and the distillation continued until a vapour temperature of 250°C . was reached. In no case was any additional water separated or observed, thereby indicating that distillation at the temperature existing in the presence of the solvent was adequate to "break" the emulsion. The

apparatus consists of an electrical source of heat, a 500 c.c. short-necked distilling flask, a special device combining the functions of distilling tube and receiver and a condenser. The source of heat may be an alcohol or gas burner, but the authors prefer a special type of electric heater. The illustration appended sufficiently describes the apparatus employed; the distilling-tube receiver is usually made by attaching a proper side-tube to a stock type of tapered graduated test-tube; cork stoppers are used for the two junctions, and if of good quality require no luting.

H. F. E. H.



Estimation of Aromatic Hydrocarbons in Petrol. D. Florentin and H. Vandenberghe. (*Bull. Soc. Chim.*, 1920, 27, 204-209.)—Twenty c.c. of the petrol are cooled in a flask immersed in cold water (or preferably ice and water) beneath a reflux condenser, and treated drop by drop, with continual shaking, with nitric acid (sp. gr. 1.5) until a brown tint is no longer produced. A few additional drops of the acid are then added, and the flask left for fifteen minutes, after which its contents are poured into a large excess of cold water, and neutralised with sodium or potassium hydroxide. Small quantities of the oxidation products of impurities in the petrol are removed by distillation in a current of steam, the petrol then decanted, the aqueous solution extracted twice with petroleum spirit, and the extracts added to the petrol. The whole is then made up to 50 or 100 c.c. with petroleum spirit, and the nitro derivatives estimated in an aliquot portion. This is evaporated over sulphuric acid under diminished pressure in a glass dish, which is weighed at intervals of ten minutes until constant in weight (or until the weight shows a constant decrease), and the amount of this residue multiplied by 0.634 gives the weight of hydrocarbons as benzene. Or the amount of nitro derivative may be estimated volumetrically by means of stannous chloride or titanous chloride, preferably the latter. For this purpose an aliquot portion of the liquid is heated with titanous chloride solution for two hours beneath a reflux condenser in a current of carbon dioxide, and the excess of the titanium salt titrated with ferric chloride solution. This method is preferable to the gravimetric method when the petrol contains constituents of higher b.pt. Examined by these methods, commercial samples of petrol were found to contain considerable quantities of benzene hydrocarbons (31.25 to 61.5 grms. per litre), and especially of toluene (fraction boiling at 100° to 130° C.). C. A. M.

Physico-Chemical Method for the Analysis of Commercial Chlorobenzene. F. Bourlion. (*Comptes rend.*, 1920, 170, 990-993.)—The sample is fractionally distilled so as to obtain a fraction boiling below 130° C. and containing the whole of the benzene with a portion of the chlorobenzene, and another fraction (the remainder) containing the rest of the chlorobenzene, together with the *p*-dichlorobenzene. The sp. grs. of the two fractions are determined, and reference to tables showing the sp. grs. of known mixtures of benzene and chlorobenzene and of *p*-dichlorobenzene and chlorobenzene gives the composition of the fractions. W. P. S.

INORGANIC ANALYSIS.

Detection of Arsenic in Sulphur. H. S. Davis and M. D. Davis. (*J. Ind. and Eng. Chem.*, 1920, 12, 479-480.)—Sulphur free from arsenic tested by Schaeppi's method (*J. Soc. Chem. Ind.*, 1881, 4, 409) gave positive tests, and the authors describe a method of their own which obviates this. The method as described by Schaeppi consists essentially in first digesting the sulphur with dilute nitric acid for the purpose of removing calcium chloride, sulphate, and sulphide. The sulphur washed free from acid is then treated with dilute ammonia solution for fifteen minutes at 70° to 80° C. Schaeppi assumes that any arsenic present in the sulphur

must exist as sulphide, mainly arsenious sulphide, and will be dissolved by the ammonia. The arsenic is then estimated indirectly through determination of the amount of sulphide by precipitation with silver nitrate in neutral solution. It was found, however, that sulphur continuously reacts with dilute ammonia at 70 to 80° C. to form sulphides, so that even arsenic-free sulphur will give a qualitative test for arsenic by this method. As is well-known, sulphur readily dissolves in hot solutions of the strong alkalis to form polysulphides, and it thus follows that a sample of arsenic-free sulphur will continue indefinitely to give a positive reaction by Schaeppi's method. The authors treat 10 grms. of the sample of sulphur with 30 c.c. of a mixture containing three parts of carbon tetrachloride and two parts bromine, and after standing for ten minutes, about 40 c.c. of strong nitric acid are added slowly in small quantities; the mixture is taken to dryness in a steam bath, and the arsenic finally determined on the residue by the Gutzeit method. The carbon tetrachloride is employed to moderate the violence of the reaction and to prevent the precipitation of sulphur.

H. F. E. H.

Separation of Chromium and Manganese. M. Herschkowitsch. (*Zeitsch. anal. Chem.*, 1920, 59, 11-12.)—The method of separation described depends on the fact that permanganate is decomposed when heated with ammonia and ammonium salts, whilst chromates are not affected. After the two metals have been oxidised to permanganate and chromate respectively, their solution is treated with ammonium sulphate and an excess of ammonia, and heated for some hours on a water-bath. The precipitate formed is collected, washed first with 5 per cent. ammonium sulphate solution, then with water, ignited, and weighed as Mn_3O_4 .

W. P. S.

Colorimetric Method of Estimating Copper and Iron in Lead and Lead Oxides. C. R. Hardy. (*Chem. News*, 1920, 120, 256.)—A weighed quantity (about 200 grms.) of the sample is dissolved in nitric acid (sp. gr. 1.2), sufficient water being added to prevent the separation of crystalline lead nitrate, and the solution treated with concentrated sulphuric acid (32 c.c. per 100 grms. of the sample). The liquid is filtered by decantation four or five times, and the filtrate treated with 10 c.c. of sulphuric acid, and evaporated nearly to dryness, cooled, diluted with water, and filtered from lead sulphate. The filtrate is evaporated to a small volume with a few drops of nitric acid, cooled, and treated with ammonia. The iron hydroxide is separated, washed, redissolved in nitric acid, reprecipitated, and thoroughly washed. The two filtrates, which will contain all the copper, are concentrated to a small volume, cooled, made up to definite volume, and 50 c.c. treated with 5 c.c. of a 0.4 per cent. solution of potassium ferrocyanide and 5 c.c. of 10 per cent. ammonium nitrate solution, and the coloration compared with that given by a standard copper solution (0.393 gm. of copper sulphate per litre). The iron precipitate is dissolved in a little nitric acid, the solution made up to 250 c.c., and 25 c.c. treated with 9 c.c. of 25 per cent. hydrochloric acid and 5 c.c. of 5 per cent. ammonium thiocyanate solution, and the coloration compared with that given by a standard iron solution (0.210 gm. of ferrous ammonium sulphate per litre) under the same conditions.

C. A. M.

Iodic Acid as a Reagent for the Microchemical Identification of Soluble and Insoluble Calcium, Strontium, and Barium Compounds. G. Denigès. (*Comptes rend.*, 1920, 170, 996-998.)—Characteristic crystals are obtained, in many cases almost immediately, when a particle of a calcium, strontium, or barium compound is treated with a small drop of 10 per cent. iodic acid solution. With calcium, the crystals have the form of sharp octahedra; with strontium, short octahedra mixed with rhombic prisms; with barium, needle-like prisms in groups. The crystals may be obtained with calcium oxalate, calcium fluoride, and strontium sulphate; barium sulphate must be reduced to sulphide (by heating on a platinum wire in a reducing flame) before it reacts with iodic acid, and calcium iodate itself must be converted into iodide by heating before the test is applied. W. P. S.

Estimation of Mercury. C. M. Bouton and L. H. Duschak. (*U.S. Dept. of the Interior, Technical Paper*, No. 227, 1920, pp. 1-44.)—A method proposed for the estimation of small quantities of mercury consists essentially in separating the metal by distillation, dissolving it in nitric acid, and titrating the solution with thiocyanate. In the case of ores or other material containing but little sulphur or organic matter, 1 grm. of the finely divided sample is mixed with 0.5 grm. of dry powdered calcium oxide, and the mixture is introduced into a combustion tube, 22 cm. long and 10 mm. in diameter, closed at one end, and into which has already been placed a layer of coarse sand. A 1 cm. layer of calcium oxide is added to the tube, and on this a like layer of sand. After a plug of brass gauze has been pushed into the tube, the latter is placed horizontally in a furnace heated at 450° to 550° C. The furnace may consist of an iron block in which a number of holes have been bored. The tube is heated for fifteen minutes, then placed open end downwards in a test-tube containing 5 c.c. of hot concentrated nitric acid, the closed end of the tube is broken off, and the acid is sucked up the tube above the ring of condensed mercury, but the acid must not be allowed to reach the gauze plug. The solution is transferred to a beaker, the combustion tube washed in a similar way with 10 c.c. of water, the total mercury solution is oxidised with permanganate, the excess of the latter destroyed with hydrogen peroxide, 2 c.c. of 10 per cent. ferric sulphate solution are added, the mixture is cooled, and titrated with standardised thiocyanate solution. If the sample contains much sulphur and organic matter, cupric oxide and a small quantity of potassium chlorate are mixed with the sample in addition to the calcium oxide, and potassium chlorate is also mixed with the layer of sand at the closed end of the tube. This procedure may be adopted for distilling mercury from precipitates collected on filter-paper, but it is better to collect the precipitate on a film of clay previously deposited in an alundum filtering crucible; the use of potassium chlorate is then unnecessary. With about 40 mgrms. of mercury there is a fairly consistent loss of 0.3 mgrm. The presence of selenium and tellurium does not interfere with the method, and arsenic and antimony have no effect on the results when cupric oxide is used in conjunction with the calcium oxide. If chlorides or sulphates are present in the sample, iron filings may be used as a reducing agent in the distillation, but it is preferable to separate the mercury as sulphide and subject this to distillation. A bibliography of some 124 methods for the estimation of mercury is given. W. P. S.

Gravimetric Estimation of Molybdenum as Sulphide. J. Sterba-Böhm and J. Vostrebal. (*Zeitsch. anorg. Chem.*, 1920, 110, 81-103.)—Molybdenum when in the form of molybdate may be quantitatively precipitated as molybdenum sulphide, MoS_3 , by means of hydrogen sulphide in the presence of a sufficient quantity of formic acid, but any reduced molybdenum present should first be oxidised by means of nitric acid. The molybdate solution (0.1 to 0.3 grm. of molybdenum) is diluted with water to 200 to 300 c.c., and treated with sufficient formic acid to give an acidity of 5 per cent., whilst if the solution contains too small a quantity of electrolytes it is advisable to add from 0.5 to 0.75 per cent. (calculated on the volume of the liquid) of pure sodium chloride to prevent the formation of a colloidal solution. Immediately after the addition of the formic acid a rapid current of acid-free hydrogen sulphide is introduced, and after thirty minutes the temperature of the liquid is raised to 40° to 50° C., and the current of the gas continued for one to one and a half hours. Or the molybdate solution may be rendered alkaline with ammonia or potassium hydroxide, and the current of hydrogen sulphide passed through it for two hours, after which it is treated with formic acid, and heated for an hour on the water-bath. The molybdenum sulphide precipitate, obtained by either method, is washed by decantation, transferred to a Gooch crucible, and dried in a current of carbon dioxide at 100° C., and subsequently at 250° C., cooled in the current of gas, and weighed in a closed weighing-bottle. C. A. M.

Method for the Estimation of Sulphur in Oils. C. E. Waters. (*J. Ind. and Eng. Chem.*, 1920, 12, 482-485.)—The author had previously worked out a method in which the total sulphur was estimated in rubber goods by decomposing that material with concentrated nitric acid saturated with bromine; the residue was then completely burned by heating with sodium and saltpetre. He now applies this method to the estimation of sulphur in oil. From 0.5 to 1.5 grms. of the oil are weighed in a 100 c.c. porcelain crucible, and 5 c.c. of concentrated nitric acid saturated with bromine are then added, a watch-glass covering the crucible; the crucible is placed on the steam-bath and carefully digested for half an hour or more; after cooling, 10 or 12 grms. of anhydrous sodium carbonate are added in small portions, the whole being thoroughly mixed with a glass rod; the resulting pasty mass is spread on the sides of the crucible, to which it should adhere, after which it is gently ignited. If more than 1 gm. of oil has been taken, there is a tendency for it to burn too briskly, but this is controllable by judicious use of the stirring-rod. It is never necessary to heat the crucible to redness. After cooling, the crucible and its contents are placed in a beaker and extracted with about 150 c.c. of water, filtered, and the sulphuric acid precipitated with barium sulphate in the presence of hydrochloric acid in the usual manner. Control experiments should be run with the reagents, and it is advisable to use a petroleum ether flame as a precaution against the presence of sulphur in gas. Duplicate analyses differ by less than 0.05 per cent., and the method has been compared with the bomb calorimeter and several other methods. The author considers it fair to claim his procedure is sufficiently accurate for all ordinary purposes, although it has not yet been applied to kerosene or gasoline. He admits that the conditions under which the barium

sulphate is precipitated are far from ideal, involving as they do the occlusion of sodium salts; of these, the sulphate tends to low results, while the nitrate has an opposite effect, and only by chance will these errors cancel one another. Estimations made with double the usual amounts of reagents go to show that the net error due to occlusion is small.

H. F. E. H.

Separation of Tin and Antimony and Estimation of Tin by Means of Cupferron. A. Kling and A. Lassieur. (*Comptes rend.*, 1920, 170, 1112-1114.)—McCay's method of separating tin from antimony by precipitation with hydrogen sulphide from a hydrofluoric acid solution (*ANALYST*, 1909, 34, 244) gives accurate results, and may be used for the estimation of the tin as stannic oxide after precipitation with cupferron. The antimony sulphide is dried at 300° C. in a current of carbon dioxide and weighed. The filtrate from the antimony sulphide is treated with boric acid (10 grms. for about 0.2 gm.), which effects a partial precipitation of stannous sulphide, and the liquid is then boiled with hydrogen peroxide to oxidise the stannous sulphide. The resulting clear liquid is cooled, and treated with a 10 per cent. solution of cupferron, and the precipitate washed with cold water, dried, ignited, and weighed as stannic oxide.

C. A. M.

Analysis of Tungsten Ores. J. Erlich. (*Ann. Chim. anal.*, 1920, 2, 102-103.)—From 1 to 2 grms. of the sample are heated for two hours on a sand-bath with 30 c.c. of 30 per cent. ferric chloride solution and 100 c.c. of hydrochloric acid; ammonium nitrate is then added, and the insoluble portion collected on a filter, ignited, and weighed. This portion is fused with sodium bisulphate and the mass dissolved in ammonia; the solution is oxidised with bromine, and elements other than tungsten are precipitated by the addition of ammonium carbonate. The precipitate is collected, ignited, and weighed. The difference between the two weights gives the weight of tungstic oxide (WO_3) present. In some cases the fusion with sodium bisulphate must be repeated to effect complete separation of the tungsten.

W. P. S.

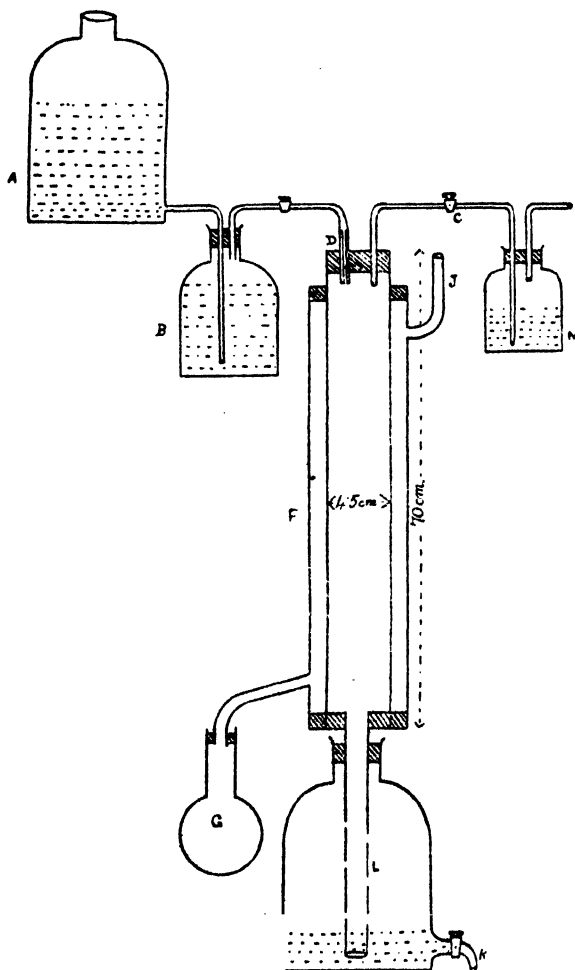
Estimation of Uranium. R. Schwartz. (*Helv. Chim. Acta*, 1920, 3, 330-346.)—Precipitation with ammonium sulphide or ammonia affords the most trustworthy means of estimating uranium; the precipitation should be made at 100° C., and in the presence of ammonium chloride. The precipitate should be ignited to U_3O_8 in a porcelain crucible, with free access of air; reduction of the U_3O_8 to UO_2 is unreliable (*cf. ANALYST*, 1902, 27, 28). Precipitation of uranium by means of ammonium phosphate from an acetic acid solution is untrustworthy. Accurate results may be obtained by dissolving the U_3O_8 in sulphuric acid and titrating the solution with permanganate solution; uranous and uranyl sulphates are formed, and the former is titrated. Uranium may be separated from aluminium by precipitation, at 70° C., with an excess of ammonium carbonate in the presence of ammonium chloride; the precipitated aluminium hydroxide must be dissolved in hydrochloric acid and the precipitation repeated in order to separate all the uranium. To separate uranium from chromium, the latter should be converted into chromate by means of

ammonium persulphate, and the uranium then precipitated with ammonia, but the precipitation must be repeated several times. By heating U_3O_8 for eight hours at 1122°C . in an atmosphere of carbon dioxide, an oxide having the composition U_2O_5 may be obtained.

W. P. S.

APPARATUS, ETC.

New Hydrogen Sulphide Generator. B. D. Steele and H. G. Denham. (*J. Chem. Soc.*, 1920, 117, 527-529.)—In the apparatus here shown, rapid evolution of the gas and complete neutralisation of the acid are effected. The steam-jacketed



tube *F* is packed with lumps of iron sulphide, and the acid holders *A* and *B* charged with dilute (1 : 1) hydrochloric acid. Steam generated in the flask *G* is passed through the jacket until the sulphide is thoroughly hot, and the tap *C* opened so that the acid drips into the heated column through the capillary tube *D*. Each incoming

drop reacts immediately, and the acid is completely neutralised long before it reaches the bottom of the column, and falls into the reservoir *H* (25 litres in capacity), the displacement of air in the latter being facilitated by having a number of holes in the tube *L*. By attaching a short air-condenser to the side tube *J*, the condensation of the escaping steam is practically complete.

C. A. M.

Falling Sphere Viscosimeter. W. H. Gibson and L. M. Jacobs. (*J. Chem. Soc.*, 1920, 117, 473-478.)—In the form of apparatus shown in the diagram various errors associated with this type of viscosimeter are taken into consideration. The tube is 29 cm. long, with an internal diameter of 2 ± 0.05 cm., and is divided into three lengths of 5 cm. for measuring the time of fall, and another 5 cm. for the ball to acquire its maximum velocity, whilst there is a graduation mark to determine the height of the liquid in the tube. The steel sphere (0.15 cm. in diameter) is delivered into the centre of the tube through a glass tube 7 cm. long and 3 mm. in internal diameter, which passes through the centre of a rubber stopper, and is adjusted to dip 3 cm. below the surface of the liquid. Applying the corrections for wall and end effects, Stokes' equation becomes—

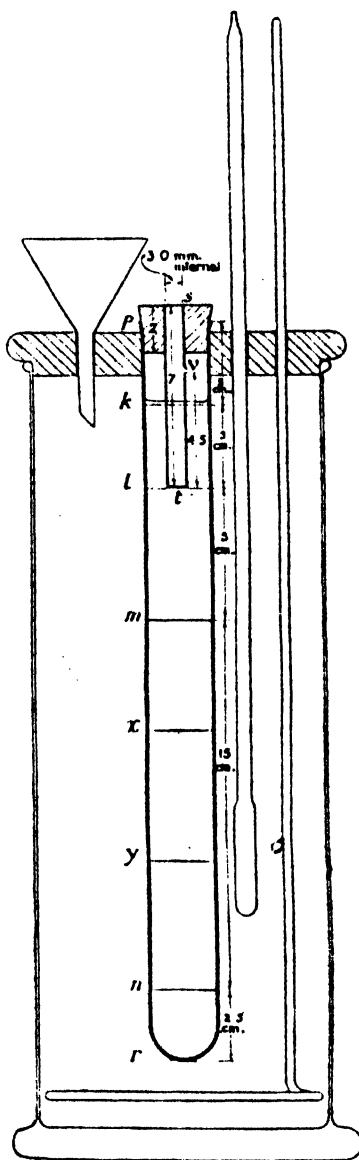
$$9\eta s(1 + 2.4x)\left(1 + 3.3\frac{r}{h}\right) = 2gr^2(s - \sigma)T,$$

where *T* represents the time of fall through a length *s*, *r* the radius of the sphere, and *h* the height of the liquid. If spheres of equal radius are used in tubes of the same dimensions, the viscosity of a liquid may be obtained by the equation—

$$\eta = \frac{(s - \sigma)T}{(s - \sigma)T_1},$$

when the time of fall for another liquid of known viscosity has been determined. The viscosity of castor oil ($\eta = 9.888$) determined by means of this instrument agreed closely with the value found by Kahlbaum and Raber.

C. A. M.



Theory of Gravimetric Analysis, with Special Consideration of Sources of Error. L. Moser. (*Zeitsch. anal. Chem.*, 1920, 59, 1-10.)—A discussion dealing principally with sources of error in gravimetric analysis. Errors due to solubility of

precipitate, induced precipitation, formation of solid solutions, absorption, and occlusion cannot be entirely eliminated, but those caused by impurities, action of gases (atmospheric oxygen, or carbon dioxide), mechanical losses, volatility, etc., may be avoided. In many cases the errors have a compensating effect. W. P. S.

Flask for the Distillation of Frothing Liquids. E. Lenk. (*Chem. Zeit.*, 1920, 44, 330.)—In the distillation of certain liquids under reduced pressure, frothing takes place at the beginning of the operation. To avoid loss due to frothing, the lower end of a vertical reflux condenser is sealed into the side of the neck of an ordinary distillation flask at a point below and opposite to the usual side tube; the latter connects with a condenser and receiver. At the commencement, the air in the apparatus is exhausted through the reflux condenser; any froth formed rises in this condenser (this has a bulbed inner tube), breaks in the latter, and the liquid returns to the flask. When frothing ceases, the tap of the reflux condenser is closed, and the distillation proceeded with in the usual way. W. P. S.

Substitutes for Platinum Wire in Bead and Flame Tests. C. C. Kiplinger. (*J. Ind. and Eng. Chem.*, 1920, 12, 500.)—To avoid waste of platinum wire or alloyed platinum wire, the author recommends the use of the "lead" in lead pencils for making bead tests. If not too large, the borax bead adheres firmly to the graphite, yet the cold borax glass may be readily removed by a knife; the reducing action of the carbon does not seem to interfere materially. Another device is the use of a clean iron nail, which can be easily cleaned and burned free from sodium; in general, the spectra of the iron salts do not interfere. A modification is described of a method used by Ehringhaus in which a strip of filter-paper is wetted with the solution to be tested and held in the flame, or one end of the paper is placed in the solution and the other in the flame. It is possible to eliminate the carbon flame which interferes with direct-vision work by supporting the burner horizontally on a stand and bringing the upper edge of the paper wick barely within the bottom portion of the flame; the salt solution is thus vaporised and colours the flame with a minimum consumption of paper. H. F. E. H.

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REPORTS.

MINISTRY OF FOOD: OILS AND FATS BRANCH.

Report by the Committee of Analysts on Standard Methods of Analysis of Seeds, Nuts and Kernels, Fats and Oils, and Fatty Residues.—A Committee of Analysts was appointed by the Director of Oils and Fats in November, 1918, to consider the settlement of standard methods of analysis of oil-seeds, fats and oils. The terms of reference of the Committee were as follows:

1. To determine the ingredients affecting the value of any of the scheduled articles.

2. To consider the usual limits of constants for good merchantable quality of the scheduled articles.

3. To arrange standard tests for all the scheduled constants for scheduled articles, and special tests.

The following took part in the deliberations of the Committee: Otto Hehner (Chairman), John Allan, G. H. Appleyard, E. R. Bolton, J. L. Buchanan, Alfred Smetham, E. W. Voelcker, W. Ellard Woolcott.

11, BILLITER SQUARE, E.C.,
February 26, 1919.

THE DIRECTOR OF OILS AND FATS, MINISTRY OF FOOD.

SIR,—The Committee of Analysts appointed by you have considered the questions embodied in the terms of reference conveyed to them at the meeting on November 13 last, and beg to submit herewith for the consideration of the Ministry of Food their report on the standard methods of analysis for oilseeds, nuts and kernels, fats and oils, and fatty residues. They present separately schedules of the data essential in defining the commercial value of particular oils, and of some of the standards governing "good merchantable quality." Other data will probably have to be added later.

The Committee have held fourteen meetings, and those members who have not been able to be present at the meetings have collaborated by correspondence.

I am, sir,

Your obedient servant,

(Signed) OTTO HEHNER

(on behalf of the Committee of Analysts).

Part I.—Determination of Oil in Seeds, Nuts and Kernels.

1. Extract a weighed quantity of the sample prepared for analysis with petroleum ether* in a suitable apparatus for two hours. Remove the partially extracted material, dry, grind in a mortar (grinding may be facilitated by the addition of 1 grm. of fine sand), and again extract until exhausted. If the sample contains a large percentage of oil it is advisable to grind twice, extracting after each grinding. Evaporate off the solvent, dry at 100° C., and weigh. In the case of oils liable to rapid oxidation, like linseed, drying should be conducted in an inert atmosphere.

2. The extracted oil must be free from solid matter and completely soluble in petroleum ether.

3. In the case of woolly cotton-seed special attention must be paid to obtaining an average sample. The seed must either be crushed by being passed through steel rollers and then thoroughly ground in an iron mortar, or well broken and ground in a mortar.

4. Owing to the variation both in the amount and composition of the oil in cross sections of the "meat" of copra, it is essential that the portion taken for analysis from the original sample be cut from the pieces at right angles to the rind.

* Petroleum ether, volatile without residue at or below 60° C.

The copra must therefore be reduced by shredding or grating at right angles to the rind, or small pieces must be completely shaved down.

5. In the case of castor-seed the clean seed with the shell is ground in a mortar, which is covered to prevent loss of husk. The extraction must be more prolonged than is the case with other seeds, and is, as a rule, complete in eight hours.

Part II.—Fats and Oils.

6. In the sample of fat or oil the following constituents are first determined : moisture, organic dirt, ash, fatty matter (by difference)—total 100.

7. Upon the clear filtered fatty matter the following determinations are made : acid value, saponification value, unsaponifiable matter, iodine value.

8. The estimations and determinations shall be carried out according to the methods described below.

MOISTURE.—9. A U-tube, one limb of which is an ordinary narrow-bore glass tube, and the other a tube about 3 feet long and from $1\frac{1}{4}$ to $1\frac{1}{2}$ inches in diameter (see appended sketch), is charged in its wider limb with soft blue Cape asbestos, which has been dried at 100°C . The wide limb is provided with a rubber stopper through which passes a narrow tube bent at right angles. The tube, without the stopper, accurately weighed, suspended by a wire, and about 2 grms. of the well-mixed sample of oil or fat are dropped on to the asbestos, and the whole reweighed. The stopper is then inserted, and the tube which passes through the stopper is connected with a Kipp apparatus supplying hydrogen, thoroughly dried by passage through strong sulphuric acid.

10. A U-tube, such as is used in elementary analysis, and provided with two glass taps, is filled with glass beads which are afterwards moistened with strong sulphuric acid. This tube is accurately weighed, after having previously been filled with dry hydrogen, and is then connected with the narrow limb of the asbestos-charged tube, the latter being suspended in a beaker containing water kept at a temperature about 10°C . above the m.-pt. of the fat. A slow current of hydrogen is now passed through the whole. When no trace of condensed moisture can be seen in the horizontal limbs of the oil tube and of the weighing tube, which is generally the case after one hour, the latter is weighed, then again attached, and a current of hydrogen allowed to pass for another hour. Minute traces of volatile organic matters, as a rule, produce a slight darkening of the sulphuric acid.

11. The asbestos may advantageously be used in the form of a very porous felt paper produced by pulping the asbestos and running the pulp into a centrifugal which is lined with linen.

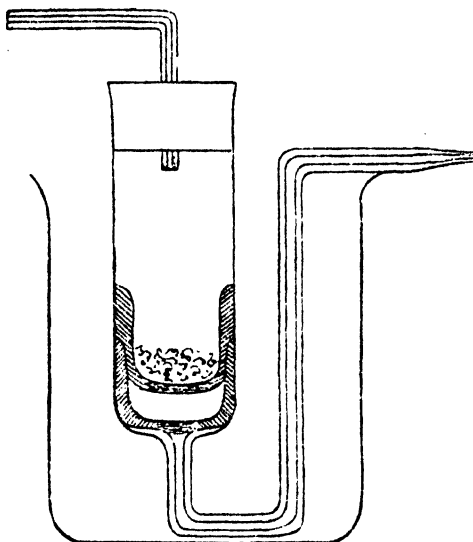
12. As an alternative method capable of dealing with a number of samples in one operation, stoppered weighing bottles of about the dimension of the wider limb of the U-tube used in the standard method may be charged with asbestos, or alternatively with strips of pleated blotting-paper or with paper pulp. These tubes are dried in a vacuum desiccator containing sulphuric acid, and kept at a pressure of 2 to 5 mm. After weighing, about 2 grms. of the fat or oil are placed in each tube, which is again weighed. The tubes are warmed till the fat is melted, and then

replaced into the vacuum desiccator. The air is exhausted and the desiccator is kept in an incubator at 20° C. for at least forty-eight hours. The weight after that time remains practically constant.

13. The standard method is applicable to all fats and oils, whether readily oxidisable, like linseed oil, or containing high percentages of free fatty acid of low molecular weight, such as lauric.

14. In the case of fats and oils which are free from uncombined fatty acid and not readily oxidisable, the ordinary methods of drying on sand in a water-oven may often be used, provided the results are in agreement with those furnished by the standard method.

DIRT AND ASH.—15. The percentage of "dirt" in a sample of oil should be arrived at by direct estimation, and not by difference.



APPARATUS FOR ESTIMATING MOISTURE IN OILS AND FATS.

16. In the case of a sample containing dirt of such a nature and size as to render it impossible to incorporate the whole of it uniformly throughout the sample, the whole of the sample should be strained through a 25 mesh wire sieve and the solid fragments picked out, weighed, and separately examined.

17. At least 10 grms. of the thoroughly mixed fat or oil is treated with about ten times its volume of hot paraffin oil (kerosene), the insoluble matter filtered off through a weighed filter, washed with hot kerosene, then with petroleum ether. The filter is dried in the water-oven to constant weight, incinerated, and the ash weighed.

18. The percentage of ash in the strained oil is determined.

19. The ash in the insoluble matter in the oil is deducted from the ash found in the oil, and this figure is taken as "soluble mineral matter."

ACID VALUE : *Definition*.—20. The acid value of a fat or oil is a measure of the

free fatty acids present, and is defined as the number of mgrms. of potassium hydroxide required to neutralise the free fatty acids in 1 grm. of the fat or oil—i.e., parts of potassium hydroxide which are required to neutralise the free fatty acids in 1,000 parts of the fat or oil.

Chemicals Required.—21. (a) Alcoholic alkali solution, $\frac{N}{10}$ and $\frac{N}{2}$ potassium or sodium hydroxide, accurately standardised. (b) Neutral alcohol, 94 to 95 per cent. (by volume), containing 0.2 grm. of phenolphthalein per litre. This may be conveniently prepared by redistilling industrial methylated spirits free from mineral oil over sodium hydroxide, adding the phenolphthalein, and subsequently neutralising.

The Test.—22. The necessary quantity of the fat or oil is carefully weighed into a 250 c.c. flask, and about 50 c.c. of the alcohol are added. The mixture is gently boiled, well shaken to thoroughly dissolve out the free fatty acids, and titrated while still warm, and with constant agitation, with $\frac{N}{10}$ alkali till the pink colour is permanent on shaking for ten seconds. Towards the end of the titration the alkali should be added drop by drop in order to avoid excess until the end point is reached.

23. As a rule, 5 grms. of the sample will be found a suitable quantity, but in the case of refined fats or oils this should be increased to 10 grms. If more than 10 c.c. $\frac{N}{10}$ alkali solution are required for the test, it should be repeated with $\frac{N}{2}$ alkali.

Then if X = number of c.c. of $\frac{N}{10}$ solution,
 F = weight of fat or oil taken,

$$\text{Acid value} = \frac{X \times 5.61}{F}.$$

24. In order to express the acidity in percentages of oleic acid, the following formula should be used :

$$\text{Oleic acid per cent.} = \frac{X \times 2.82}{F}.$$

25. In the case of fats obtained from the kernels of the coconut group, such as coconut oil, palm, kernel oil, tucum, babassu, cohune, etc., the acidity is calculated as lauric acid, when the following formula should be used :

$$\text{Lauric acid per cent.} = \frac{X \times 2.0}{F}.$$

SAPONIFICATION VALUE: Definition.—26. The saponification value represents the amount of potassium hydroxide required to neutralise the free and combined acid constituents of a fat or oil, and is expressed in terms of parts of potassium hydroxide per 1,000 parts of the fat or oil.

Solutions Required.—27. (a) $\frac{N}{2}$ hydrochloric acid accurately standardized. (b) An alcoholic solution of potassium hydroxide or sodium hydroxide, approximately $\frac{N}{2}$, prepared by dissolving 18 to 20 grms. of stick potassium hydroxide in not more than 10 c.c. of distilled water, and making up to 500 c.c. with 94 to 95 per cent. (by volume) alcohol, which may be prepared as in par. 21 (b). The solution is allowed to stand for twenty-four hours, and the clear liquid siphoned off for use. Potassium hydroxide may be replaced by sodium hydroxide, but this substitution is not desirable. The alcohol must be free from mineral oil and of such a purity as to yield a nearly

colourless solution after twenty-four hours. (c) An alcoholic solution of phenolphthalein, 1 per cent.

The Test.—28. About 2 grms. of the clear filtered fat are accurately weighed into a 200 c.c. flask of resistance glass, 25 c.c. of neutral alcohol added, and 25 c.c. of the alcoholic potassium hydroxide solution accurately measured run in. A like quantity of the same solution is run in, in exactly the same way, into a similar flask, together with 25 c.c. of the neutral alcohol. The flasks are connected to reflex condensers and heated, preferably in a water-bath, so that the alcohol boils briskly for thirty minutes. The flask containing the fat should be shaken with a rotary movement from time to time during the period. The contents of the flasks are then titrated while hot with the $\frac{N}{2}$ acid, after the addition of 1 c.c. of the phenolphthalein solution.

If F = grms. of fat taken,

X = c.c. of acid required in the control experiment,

Y = c.c. of acid required to neutralise the excess of alkali in the test,

$$\text{Then saponification value} = \frac{X - Y \times 0.02805 \times 1,000}{F}$$

$$\text{N.B.—The saponification equivalent} = \frac{561,000}{S}$$

ACID VALUE AND SAPONIFICATION VALUE (COMBINATION METHOD).—29. The acid value and saponification value may, if so desired, be determined upon the same weight of the sample in the following manner:

30. About 4 grms. of the clear filtered fat or oil are accurately weighed into the saponification flask, and 25 c.c. of hot alcohol containing 1 c.c. of phenolphthalein solution, and previously neutralised, are added. The free fatty acids are titrated from a burette with the alcoholic alkali solution as used for the saponification value, warming if necessary during the titration. The number of c.c. required by the free fatty acids having been noted, such a further quantity of alcoholic alkali as will make a total of 50 c.c. is added, and the process continued as described above.

31. A blank test is made with 50 c.c. of the alcoholic alkali delivered from the identical burette, using similar quantities of all the reagents. The exact value of the alcoholic alkali having been determined with $\frac{N}{2}$ hydrochloric acid, the acid value and saponification value may be respectively calculated.

UNSATURATED MATTER.—32. Unsaponifiable matter in fats and oils includes all substances which are insoluble in water but soluble in the fat solvents specified below after alkali saponification of the fats and oils.

33. Saponify 5 grms. of the sample with 50 c.c. approximately normal alcoholic KOH, boiling under a reflux condenser for sixty minutes with occasional shaking. Transfer the alcoholic solution to a separating funnel; rinse the flask successively with 50 c.c. of methylated ether, 50 c.c. of petroleum ether, and 50 c.c. of water. Transfer the rinsings to the separating funnel, shaking well after each addition. Allow to separate, and withdraw the soap solution. Repeat the extraction of the soap solution twice, using ether and petroleum ether as before, omitting the treatment with water. The combined ethereal extracts are washed by shaking with slightly alkaline water, allowing to settle, and run through a filter into a tared flask. Wash the filter

with petroleum ether, distil off the solvents, dry at 100° C., and weigh the saponifiable matter.

IODINE VALUE : 34. Reagents Required : (a) *Iodine Solution*.—35. This is conveniently prepared by dissolving 7.5 grms. of iodine trichloride in acetic acid (minimum strength 95 per cent.), and solution may be hastened by warming on a steam bath. When dissolved, add to the solution 8.2 grms. of resublimed iodine, assist solution by heating as before, and make up to 1,000 c.c. with acetic acid (95 per cent.) The solution is standardised by means of a blank test carried out at the time that it is used, which should be at least twenty-four hours after it is made up. If the solution is heated for a short time by immersion in boiling water, it may be used immediately after cooling.

(b) *Sodium Thiosulphate Solution*.—36. Dissolve 24.8 grms. of the pure salt in 1 litre of distilled water. It is advisable to add 0.5 gm. per litre of sodium bicarbonate to the solution as a preservative. This solution must be standardised by titrating it against pure dry resublimed iodine, but where this is inconvenient the iodine may be liberated from potassium iodide by a known amount of potassium dichromate in the presence of hydrochloric acid. As the oxidising value of potassium dichromate does not always correspond accurately with that of an equivalent amount of iodine, the potassium dichromate to be used as described below shall first be set against pure dry iodine. The titration is carried out as follows : Weigh 0.20 gm. of pure recrystallised $K_2Cr_2O_7$ into a 200 c.c. stoppered bottle, dissolve in 25 c.c. distilled water, add 20 c.c. 10 per cent. potassium iodide solution and 10 c.c. pure HCl, moisten the stopper with the KI solution, and allow to stand five minutes. Wash down the stopper with distilled water, dilute to about 100 c.c. volume, and titrate the liberated iodine with the sodium thiosulphate solution, using starch solution as indicator. A blank test, using the potassium iodide solution and hydrochloric acid alone, should be carried out, and the necessary deduction, if any, made from the previous titration.

$$0.2 \text{ gm. } K_2Cr_2O_7 = 0.51768 \text{ iodine.}$$

(c) *Potassium Iodide Solution*.—37. A 10 per cent. solution of the pure salt in distilled water.

(d) *Solvent*.—38. Chloroform or carbon tetrachloride to be used as a solvent for the oil. A blank test on the solvent must not show an absorption of iodine equal to more than 0.2 c.c. of the thiosulphate solution.

(e) *Starch Solution*.—39. Use a 1 per cent. solution of soluble starch.

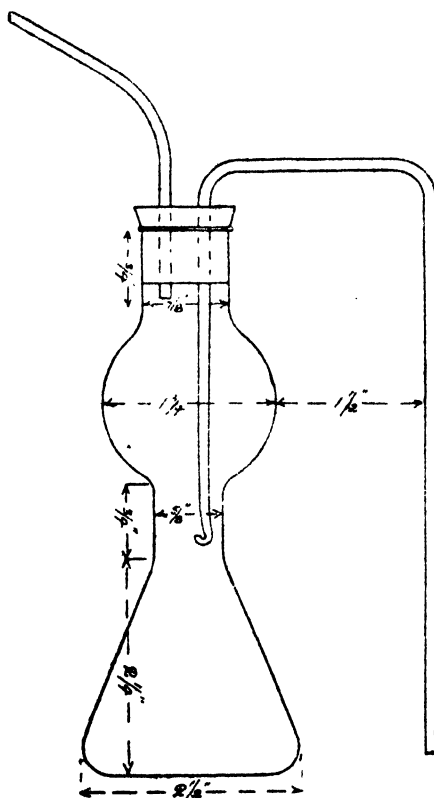
Method.—40. 0.15 to 1 gm. of the sample is weighed into a wide-necked stoppered bottle of about 200 c.c. capacity ; 0.15 gm. of a strongly drying oil and a proportionately larger amount for oils or fats of lower iodine value, up to approximately 1 gm. for such fats as coconut and the like. Dissolve the weighed quantity of fat or oil in 10 c.c. of the solvent (d) and add 25 c.c. of the iodine solution, moisten the stopper with potassium iodide solution, and allow to stand for one hour. In the case of fats having a very high iodine value like linseed, allow to stand for three hours.

41. After standing, wash the stopper and neck of the bottle down with 15 c.c. of

the 10 per cent. potassium iodide solution, mix, and add 100 c.c. of distilled water, and titrate the excess of iodine with the sodium thiosulphate solution. Towards the end of the titration add about 2 c.c. of the starch solution, and shake vigorously after each addition of the thiosulphate solution until the contents of the bottle are colourless.

42. A blank test, using 10 c.c. of fat solvent and 25 c.c. of the iodine solution, must be done with each set of estimations, or at least once daily.

43. The result is expressed as per cent. of iodine reacting with the fat.



SEPARATING FLASK

44. N.B.—Where a large number of liquid oils have to be tested it may be convenient to weigh them by dropping them from a small lipped tube and ascertaining the weight taken by difference, while solid fats can be arranged in small cubes on a suitably-sized glass plate and picked up with a pin which has been tared along with the whole, in this case also the weights of the successive pieces are determined by difference until the last piece has been weighed.

SPECIFIC GRAVITY AND VISCOSITY.—45. In cases where it is desirable to determine the specific gravity or the viscosity of an oil or fat, the specific gravity shall be determined by the ordinary standard methods and viscosity by the Redwood viscometer.

TITER TEST.—46. In cases where it is desirable to determine titer the method to be adopted is that prescribed by the Seventh International Congress of Applied Chemistry. This method is given in full in J. Lewkowitsch's "Chemical Technology of Oils and Fats," fifth edition, 1913, vol. i.

Part III.—Fatty Residues: Evaluation of Soap Stock, Acid Oils and Fatty Acids, Cotton Oil, Black Grease and Mucilage.

47. A weighed quantity of the sample (sufficient to give from 1 to 2 grms. of fatty extract) is introduced into a separating flask as illustrated, 10 to 20 c.c. of hot water are added, followed by one drop of methyl orange and a sufficient excess of hydrochloric acid to insure the decomposition of soap; 5 grms. of salt are added, and the mixture is heated gently until the acids separate in a clear layer.

48. The contents of the flask are allowed to cool to about 30° C., and 20 c.c. of petroleum ether and the liquors are gently agitated, and water is added until the junctions of the water and ether rises to the bottom of the centre constriction. When the separation is complete the ether is blown off by means of a siphon and, if necessary, filtered into a weighed flask, 10 c.c. of petroleum ether are introduced into the separating vessel, and again siphoned off. Two further portions of 10 c.c. of petroleum ether are then used, and in these latter cases the flask is closed with a stopper, and gently inverted two or three times after each addition of ether.

49. The mixed ethereal extracts are distilled and the residue dried at 100° C. and weighed.

Report by the Committee of Analysts on Standards of Good Merchantable Quality.—Members of the Committee of Analysts: Otto Hehner (chairman), John Allen, G. H. Appleyard, E. R. Bolton, J. L. Buchanan, Alfred Smetham, E. W. Voelcker, W. Ellard Woolcott.

11, BILLITER SQUARE, E.C.
March 5, 1919.

TO THE DIRECTOR OF OILS AND FATS, MINISTRY OF FOOD.

SIR,

1. In the terms of reference submitted to us by you at our first meeting on November 13, 1918, we were asked to arrange standard tests for all constants and all schedules for oil seeds, nuts and kernels, and oils and fats. We have already communicated to you the results of our deliberations on this point.

2. We now forward for your consideration our report on the two other terms of reference which we have interpreted as follows: (i.) To determine the ingredients affecting the value of any of the scheduled articles; (ii.) to consider the usual limits of constants for good merchantable quality of the scheduled articles.

3. **STANDARDS FOR VEGETABLE OILS.**—An official schedule for vegetable oils, home manufactured, good merchantable quality, was submitted to us embodying a blank schedule for the insertion of standards and constants under several headings. After due consideration we decided that although all of these particulars are of interest to analytical chemists it would not be practically useful to insert the whole

of these in a contract. We therefore decided to limit our recommendations to those characteristics which have an immediate bearing on the commercial value of the oil.

4. We recommend the following standards for oil of "good merchantable quality" on the understanding—(i.) that such oils shall be free from foots and foreign matter (a mere compliance with the standard prescribed does not furnish proof of purity). (ii.) That an allowance should be made to the buyer or the seller for any deviation from these standards.

The figures are based on records covering, in some cases, a considerable period of years, and have been selected as representing a fair average, and as tending to raise rather than lower the existing standard.

5. We realise that other considerations actually enter into the value of different oils, specially those required for edible purposes. There is at present a very great difficulty in defining systematically such qualities as colour, turbidity and odour, but it might be possible eventually to secure such a definition.*

6. SEEDS, NUTS AND KERNELS ("GOOD MERCHANTABLE QUALITY").—We have also considered the definition of "good merchantable quality" in the case of seeds, nuts and kernels. In this case, too, we have selected the figures from records which different firms have been kind enough to make available, and have sought to establish an average which will tend to the general improvement of the product. In some cases, where the same kind of produce is imported from widely differing sources, there is necessarily a good deal of variation. Moreover, produce coming from the same source is liable to differ in standard from year to year, and at different periods of the same year.

7. We have, nevertheless, come to the conclusion that it would be a great advantage to have standards that might be applied with an allowance, over or below, to the seller or to the buyer, for all kinds of produce. We think that such standardisation, even in the case of crops varying so much as cotton-seed and linseed from place to place and year to year, would tend to the improvement of the produce, and to better conditions of storage and transport. We have therefore given standards for one or two classes with a view to simplification.

8. The standards which we suggest are calculated on the pure produce, and assume that the determination of shell and dirt has already been carried out. The question whether the oil content of any oleiferous admixture which is not pure produce should be estimated depends on the terms of the contract, and is a commercial matter on which we are not competent to offer a recommendation. As, however, impurities are usually the same kind from the same country of origin, it might be possible to fix an average oil content, if this were required, for the foreign oleaginous admixture.

9. The data in the schedule which we propose conform to those already adopted for the West African Contract, but we have included data for other seeds, nuts and kernels which are not covered by that document. The schedule is as follows :

* We understand that a Sub-committee of the Oils and Fats Committee of the Food Investigation Board are now considering the question of palatability of edible oils, and that further research into the general question is in contemplation.

SCHEDULE.

(Oil of "good merchantable quality" shall be free from foots or foreign matter, and shall conform to the following standards.)

Oil.	Percentage of Free Fatty Acids (Oleic, except where otherwise stated).	Percentage of Moisture not exceeding—	Percentage of Unsaponifiable Matter.	Other Standards.		
LINSEED OIL—				Iodine value (Wijs) 185.		
Raw	2	0.25	1			
Refined	2	0.25	1			
COTTON OIL—						
Crude, Indian ...	2.5	0.25	1.5	{ Specific gravity, 0.915. Viscosity, Redwood standard.		
„ Egyptian ...	4					
Technical } ...	0.3					
Refined } ...	0.3					
Common edible ...	0.3					
Fine edible ...	0.15					
GROUND-NUT OIL—						
Crude	4	0.25	1			
Fine edible ...	0.15					
SESAME OIL—						
Crude	3.5	0.25	1			
Fine edible ...	0.15					
RAPE OIL—						
Crude	1	0.25	1			
Technical } ...	1					
Refined } ...	0.1					
Fine edible ...	0.1					
PALM-KERNEL OIL—						
Technical	5 lauric	0.25	1			
Fine edible ...	0.1 „					
CASTOR OIL—				Specific gravity, 0.963.		
Pharmaceutical ...	1	0.25	1			
Firsts	1					
Seconds	4					
SOYA OIL—						
Crude	2.5	0.25	1			
Fine edible ...	0.15					
MAIZE OIL—						
Crude	3	0.25	2			
Fine edible ...	0.15					
COCONUT OIL (Technical)—						
Cochin quality ...	1	0.25	1			
Ceylon	2					
Copra	4					
Fine edible ...	0.1					

SCHEDULE OF STANDARDS FOR SEEDS, NUTS AND KERNELS.

	Oil Content.	Percentage of Free Fatty Acids* in Extracted Oil.
Babassu	66	2 (lauric).
Castor-seed	47	1
Copra	66	2 (lauric).
Cotton-seed, Egyptian type	22	3
" Indian " 	18	2
Benni-seed, gingelly or sesame	54	3
Decorticated ground-nuts	48	6
Undecorticated ground-nuts	37	2
Linseed	38	1.5
Mowrah	58	—
Palm-kernel	52	6 (lauric).
Rape-seed	43	2
Shea-nuts	46	10
Soya	17	2

* Calculated as oleic unless otherwise stated.

10. ANALYSIS OF FEEDING STUFFS.—The method of analysis of oil-cake is prescribed under the Fertilisers and Feeding Stuffs Act. As the use of petroleum ether as a solvent has been agreed upon for oleaginous seeds, nuts and kernels, we should like to suggest that the Board of Agriculture be invited to consider the use of the same solvent for oil-cake on the ground that ether at present prescribed as the solvent has a tendency to extract from oleaginous materials non-fatty substances.

11. The schedules referred to us did not include animal fats, but we are willing to investigate these if desired.

We are, Sir,

Your obedient servants,

(Signed)

(The Committee of Analysts.)

* * * * *

REVIEWS.

QUANTITATIVE ANALYSIS BY ELECTROLYSIS. By ALEX. CLASSEN, with the co-operation of H. CLOEREN. Revised, rearranged, and enlarged English edition by W. T. HALL. Pp. xiii + 346. New York: J. Wiley and Sons, Inc.; London: Chapman and Hall, Ltd., 1919. Price 17s. 6d. net.

The appearance of a new English edition of Classen's well-known book will be generally welcomed. The present edition is a revision by W. T. Hall of a translation of his which was published about six years ago.

To the majority of English readers Classen's book is probably known from B. B. Boltwood's translation of the fourth German edition, and to these the present volume will appear practically as a new book. The theoretical introduction has been entirely rewritten. It occupies about a third of the book, and gives a good account of the subject. The apparatus is clearly described, and details of most of the methods of rapid electrolytic analysis are supplied, the apparatus in use in the author's laboratory being more fully dealt with. In the practical part it will be found that in agreement with the results of modern work on the subject simple electrolytes have, wherever possible, been given the preference over complex ones. The methods of determination and separation of metals based upon the control of the potential of the cathode are dealt with both in the theoretical and the practical part. The processes emanating from Edgar F. Smith's laboratory for the determination and separation of the metals of the alkali and alkaline earth groups and for the simultaneous estimation of anions have been included. A special part is devoted to the analysis of commercial products. The work of the translator has been well performed throughout, and several additions have been made by him. Thus in the practical part a number of new methods will be found, and the more important data, such as equilibrium and over-voltage, are stated at the head of the section dealing with each element. In the theoretical part the history of electrolytic analysis has been extended by a short account of the history of theoretical electrochemistry, in which the modern views of the structure of the atom and of the process of ionisation are discussed. A theoretical chapter has also been added dealing with electrometric titrations and the applications of potential and conductivity measurements; the matter is, however, not again referred to in the practical part. The book may be thoroughly recommended to technical and teaching laboratories.

H. J. S. SAND.

THE CHEMISTRY OF COAL. By JOHN B. ROBERTSON. London: Gurney and Jackson, 1919. Price 3s. 6d. net.

This little monograph is divided into two distinct sections; in the first it has been the aim of the author to show the chief methods by which the problems of coal constitution have been attacked, and to outline some of the more interesting results obtained. In the first five chapters a useful summary is given of the theories of coal formation and the work which has been done by the aid of solvents, by oxidation,

and by destructive distillation, with a view to throwing light on the constitution of coal.

Chapters VI. to IX. deal with sampling, analysis, and calorimetry of coal. There is nothing novel in this section, but the methods are well described, and the author has found them successful in practice, and attention is drawn to sources of error which are sometimes overlooked. The calorimetric bomb method is described very clearly, but exception must be taken to the author's statement that the *technical heat value* differs from the determined calorific value by the latent heat of the steam condensed to water in the bomb. The result so obtained (the net value) is a mere convention; it takes no account of the sensible heat in the water vapour escaping at flue-gas temperature in practice—an amount different in every boiler plant. In his concluding chapter he deals briefly with "The Properties of Coal on Combustion." Altogether the book is well written, and is a useful little addition to the books on coal.

J. S. S. BRAME.

FUEL PRODUCTION AND UTILISATION. By H. S TAYLOR, D.Sc. London: Baillière, Tindall and Cox, 1920. Price 10s. 6d. net.

Dr. Taylor has certainly produced a book which, as he states in his preface, shows points of divergence from the standard textbook on "Fuel." He is modest in his claim, for his book is conceived on quite different lines to the ordinary textbook, and has been admirably arranged. The textbook, on orthodox lines, serves a most useful purpose; it is indispensable to the ordinary student, and Dr. Taylor's book as a supplementary treatise on fuel dots the i's and crosses the t's. It is largely compiled from the valuable publications on fuel resources and their efficient utilisation which have been issued by the United States and the Canadian Bureau of Mines, and from other authoritative sources.

One may safely say that every phase of the fuel problem receives adequate treatment, particularly the minor fuels and the utilisation of waste fuels. These may appear to be more fully treated than their present importance warrants, but in view of their undoubted future importance, with the great rise in price of the more staple fuels, this full discussion is to be welcomed.

The chapter on synthetic fuels, which concludes the volume, will be found of very considerable interest, dealing as it does with acetylene, "the synthesis of liquid hydrocarbons from coal," synthetic alcohol and benzol. Hexamethylene (cyclohexane), referred to in an earlier chapter, might well have been included here.

Dr. Taylor has in many cases drawn at considerable length from his authorities, but his selection of material has been excellent, and it is no disadvantage to have the statements in the words of the responsible authors. In one direction only has the author made what may be regarded as a mistake, and that is in sometimes quoting costs and estimates based on pre-war figures. Unfortunately, prices like one shilling per thousand cubic foot for coal-gas and the same price for a gallon of petrol are to-day but reminders of a happy state of things which existed "once upon a time"—a phrase we learn in childhood to associate with fairy tales!

J. S. S. BRAME.

MANUAL OF THE CHEMICAL ANALYSIS OF ROCKS. By HENRY S. WASHINGTON, Ph.D. Third Edition, revised and enlarged. 1919. New York: John Wiley and Sons, Inc. London: Chapman and Hall, Ltd. Price 11s. 6d. net.

This well-known work now appears in its third edition. It describes in detail the analysis of a typical silicate-rock, and the methods employed will give results as accurate as is possible with our present-day knowledge. The introduction is followed by a general discussion on apparatus and reagents, sampling, and methods. Then the procedure needed for determining the individual constituents is treated in detail. These methods can now be regarded as having been more or less standardised by use.

There are many traps in the analysis of silicates, and it is only with the confidence of ignorance that these analyses have been undertaken by skilful analysts unprepared for the peculiar errors to which the analyses are susceptible. The $100 \pm x$ per cent. total is not necessarily a satisfactory check, since if one constituent is high, another will be proportionally low. As a result, a comparatively large percentage of published analyses, even where conscientiously performed, are altogether useless. Here are two recent analyses of the same dried clay reported upon commercially by two independent men. The figures are rounded to the first decimal :

	SiO ₂ .	TiO ₂ .	Al ₂ O ₃ .	Fe ₂ O ₃ .	MgO.	CaO.	K ₂ O.	Na ₂ O.	Loss on Ignition.
(1)	73.6	—	12.5	4.1	0.04	0.01	2.12		7.7
(2)	51.9	1.1	17.5	8.4	4.4	3.2	3.9	1.4	7.8

Here is another pair of analyses of another dried clay reported by the same two independent sources :

	SiO ₂ .	TiO ₂ .	Al ₂ O ₃ .	Fe ₂ O ₃ .	MgO.	CaO.	K ₂ O.	Na ₂ O.	Loss on Ignition.
(3)	63.0	—	6.8	3.9	3.2	5.6	1.9		15.6.
(4)	35.3	0.6	14.4	3.7	8.3	12.9	3.5	0.9	20.2

The writer can guarantee that analyses (2) and (4) were conscientiously performed, but the differences between (1) and (2) and (3) and (4) are so great that it is inconceivable that even Washington's book would be of any assistance to one of them, for these numbers must have been obtained by a parasite on the profession—namely, the *guessing analyst*.

Although silicate analyses can be conducted with a high degree of accuracy, the methods unfortunately occupy an inordinately long time—four or five days. A great deal of platinum ware also is needed for the operations. This all accentuates the cost of a commercial analysis. The cost of platinum for chemical and other important work is now so high that it is time the demand for this indispensable metal was relieved by prohibiting its use in the manufacture of jewellery for ornamenting the fair sex.

J. W. MELLOR.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE EFFECT OF PRESSURE UPON THE POLENSKE AND REICHERT- MEISSL VALUES.

By VINCENT H. KIRKHAM, F.I.C.

GENUINE butter fats analysed in the Government Chemical Laboratories, Nairobi, British East Africa, invariably give very low Polenske values, and the reason for this was made the subject of research.

When considering the explanation of the phenomenon, one of the first ideas to suggest itself was the possibility of the low atmospheric pressure affecting the composition of the distillate. Nairobi is situated at an altitude of about 5,500 feet above sea-level; the barometer generally read about 627 mm., and water boils at roughly 95° C.

In order to test this theory it was only necessary to devise a means of performing the Reichert-Meissl-Polenske distillation under different pressure conditions, and this was effected by attaching a Brühl jar to the receiver end of the condenser, and attaching to this jar a manometer, a bicycle valve and pump, and a Geryk vacuum pump.

All corks were carefully wired down and the lid of the Brühl jar secured, when pressures above that of the local atmosphere were required, by the use of clamps.

The only part of the standard apparatus which it was necessary or desirable to alter was the substitution of a 300 c.c. round-bottomed Jena flask, with neck cut down to the prescribed length, for the flat-bottomed flask normally used.

The butter fat used in these experiments was obtained from butter made in the laboratories.

Strict adherence to the prescribed conditions was observed in carrying out the observations, and it is unnecessary to describe here such a well-known process. The only points worth mentioning are that the times taken for distillation were a little over the twenty minutes—generally twenty-five minutes—that at very low pressures much difficulty was experienced in maintaining steady ebullition, and that one deviation from the usual procedure was made—*i.e.*, the 110 c.c. distillate was agitated and allowed to remain a few hours before filtration and titration. The reason for this

deviation is that a perfectly limpid filtrate is not obtained often by the usual rapid method, with the probable result that a trace of insoluble acid is passing into the filtrate.

The experimental results obtained were as follows :

Pressure. Mm.	Reichert-Meissl Value.	Polenske Value.
100	22.34	0.19
180	24.43	0.48
250	25.57	0.75
380	26.93	1.14
450	27.13	1.61
627	27.60	2.06
760	27.99	2.68
900	28.17	—
1,000	28.05	3.40

If these values are plotted against the pressures—and it will be seen at once that there is a considerable difference in the effect of pressure upon the Polenske and Reichert-Meissl values—the Polenske value increases steadily with the pressure, and the following empirical formula represents the relationship: $V = \frac{v(P - k)}{p - K}$, when P = pressure at which Polenske value is V ; p = pressure at which Polenske value is v ; K = constant, or pressure at which Polenske value is zero, in this case 45.

Therefore Polenske corrected to normal pressure = $\frac{\text{observed Polenske} \times (760 - 45)}{(\text{barometric pressure} - 45)}$.

The variations in barometric pressure in any one locality may well lead to errors up to 10 per cent. of the value. In the case of laboratories situated at high altitudes the errors may exceed 25 per cent. Records made during seasonal depressions in the atmospheric pressure would, unless corrected, lead to the erroneous deduction that there was a seasonal depression in the Polenske value of the fat. In calculating adulteration the error might be very great and lead to serious consequences.

Pressure. Mm.	Reichert-Meissl.		Polenske.	
	Found.	Calculated.	Found.	Calculated.
100	22.34	22.58	0.19	0.19
180	24.43	24.19	0.48	0.48
250	25.57	25.10	0.75	0.78
380	26.93	26.23	1.14	1.19
450	27.13	26.69	1.61	1.44
627	27.60	27.60	2.06	2.07
760	27.99	28.12	2.68	2.55
900	28.17	28.60	—	—
1,000	28.05	28.87	3.40	3.40

In the case of the Reichert-Meissl curve the diminution of effect with increasing pressure suggested a logarithmic function, and trial was made of the following formula :

$$V = \frac{(v-K) \log P}{\log p} + K, \text{ when } K=10,$$

and although the agreement is not very close it enables a correction to be applied which will reduce the error to a negligible amount.

The agreement between the formulæ and the experimental determinations is shown in the table.

THEORETICAL CONSIDERATIONS.

That such simple empirical formulæ can express the relationship between pressure and Polenske and Reichert-Meissl values might lead one to suppose that some equally simple laws were involved. It is disappointing to find that no such simple explanation is forthcoming, and that the simplicity of the result under the complexity of the various factors involved in the performance of a Reichert-Meissl-Polenske distillation appears to be fortuitous.

The fundamental difference between the Polenske and Reichert-Meissl variations with pressure may be attributed to the fact that, as Regnault showed, non-miscible liquids vaporise independently of each other, and the proportions of each in the vapour phase have no relationship to the proportions present in the liquid phase, but are entirely dependent upon the ratio of their partial pressures and vapour densities,

$$\frac{w}{W} = \frac{d.p.}{D.P.} \text{ (Nauman),}$$

whereas in the case of miscible liquids the proportions of each present in the vapour phase have a relationship with the proportions in the liquid phase, but that relationship is greatly affected by attractions between like and unlike molecules. In the simplest case, the relationship may be represented thus :

$$\frac{x_1}{x_2} = C \frac{W_1}{W_2} \text{ (Brown),}$$

where x_1 and x_2 are the fractions vaporised, W_1 and W_2 the masses in the liquid phase, and C a constant not differing much from the ratio of the vapour pressures of the substances at the temperature of distillation. But this simple relationship between the composition of the vapour and liquid phases is only true of miscible liquids of closely related constitution, or of other mixtures when

$$P = \frac{mP_a + (100 - m)P_b}{100} \text{ (Zawidski),}$$

and in the case of fatty acids and water the relationship does not hold good, particularly as butyric acid forms a maximum vapour pressure binary mixture.

To deduce the effect of pressure upon the proportions of soluble and non-soluble acids in the distillate from a knowledge of vapour pressures becomes hopelessly involved when it is remembered that in the R.M.P. mixture there exist—(1) water; (2) glycerol; (3) fatty acids of very low vapour pressures (non-volatile); (4) fatty acids of vapour pressures sufficient to yield a measurable mass in the distillate, (3) and (4)

being insoluble or only slightly soluble in water; (5) fatty acids of high vapour pressure, soluble in water.

The presence of the glycerol will reduce the partial pressure of the aqueous vapour, and its effect upon the partial pressures of the fatty acids will depend upon their solubility in glycerol. The fatty acids will constitute a series of miscible liquids, the relative proportions of which will affect the partial pressures of each constituent; while the non-miscibility, partial or complete miscibility, of the individuals of the series with water make the problem extremely complicated.

Molecules of all the fatty acids will occur in the distillate, and if the series form a mixture comparable with simple mixtures where

$$P = \frac{mP_a + (100 - m)P_b}{100},$$

then it is obvious that the proportions of the various fatty acids occurring in the liquid phase will affect the proportion occurring in the vapour phase (distillate), even though these various acids are not soluble in water. In other words, the Polenske acids must comprise oleic, stearic, palmitic, myristic, lauric, and probably capric (which has failed to dissolve in the R.M. acid and water) acids, in proportions depending to some extent upon their proportions in the liquid phase.

I am indebted to Prof. Sydney Young for pointing out that in the particular case of myristic acid and water the ratio of the partial pressures does actually increase in proportion to the total pressure when a simple mixture of these two substances is subject to experiment, but that with lauric acid the ratio of the partial pressures does not keep pace with the increase of total pressure. In my experiments it has been seen that the ratio of the partial pressures of the Polenske acid to that of water has increased in proportion to the increase in total pressure, precisely as would occur if water and myristic acid were distilled. The composition of the Polenske acids is not well known, but the composition of butter fat is as follows (Brown):

Glycerides of—				Per Cent.
Dihydroxystearic acid	...	...	...	1.04
Oleic acid	...	...	...	33.95
Stearic acid	...	...	...	1.91
Palmitic acid	...	...	...	40.51
Myristic acid	...	...	...	10.44
Lauric acid	...	...	...	2.73
Capric acid	...	...	...	0.34
Caprylic acid	...	...	...	0.53
Caproic acid	...	...	...	2.32
Butyric acid	...	...	...	6.23

The composition of the Polenske acids might be roughly deduced from a consideration of the vapour pressures of the above acids at the temperature of distillation and the molecular proportions in which they occur. The vapour pressure of lauric acid is something like two and a half times that of myristic acid, but there is present nearly four times as much myristic as lauric acid. The large proportion of palmitic acid must also contribute its quota to the Polenske acids, and in this case it is probable that the ratio of partial pressure of the acid to that of water would exceed

the ratio of total pressure, but, as was stated above, the behaviour of a mixture of a number of miscible substances cannot be predicted with accuracy, and it is remarkable that the experimental results should show that the mixture present in the R.M.P. process with a normal butter fat yields results at different pressures of such a simple character, and that these results should agree with what would occur if water and myristic acid alone were present.

As regards the Reichert-Meissl (soluble volatile acids), we may assume that they consist principally of butyric, caproic, caprylic, and possibly a portion of the capric acids.

It is difficult to predict anything from a knowledge of vapour pressures. Butyric acid forms a mixture of maximum vapour pressure of b.pt. 99.2° (760 m.) containing 20 per cent. butyric acid (*vide* Young, "Fractional Distillation," p. 67). From this it might be expected that from 145 c.c. aqueous solution yielding 110 c.c. distillate the whole of the butyric acid would be obtained at most temperatures, and that the increase in R.M. figure at higher pressures and temperatures was to be attributed more to the increase in caproic and caprylic acids. It is unfortunate that the Kirschner figures were not obtained at the same time.

As was stated before, the relationship between the composition of vapour and liquid phases does in its simplest case approximate to

$$\frac{x_1}{x_2} = C \frac{W_1}{W_2},$$

when C is a constant not dissimilar to the ratio of the vapour pressures of the substances at the temperature of distillation. It is noteworthy that the ratio of the partial pressures of water and the lower fatty acids does not increase so rapidly as the temperature (and therefore total pressure) increases, as is the case with the higher fatty acids, and this fact alone explains why the R.M. figure cannot increase to the same extent as the Polenske when the distillation is performed at higher pressures (and temperatures), but no basis for a theoretical deduction of the empirical formula found by experiment exists.

CONCLUSIONS.

1. The Polenske value is a function of the pressure, and unless values are corrected to normal pressure serious errors are liable to be introduced.

With butter fat the relationship is as follows:

$$V = \frac{v(P - K)}{p - K}.$$

2. The Reichert-Meissl value is a logarithmic function of the pressure, and the errors introduced by ordinary variations in atmospheric pressure are quite small.

With butter fat the relationship is as follows:

$$V = \frac{(v - K) \log P}{\log p} + K.$$

Experiments are being conducted to ascertain the partial pressures of the several fatty acids with water, the partial pressures of various mixtures of acids and water, and the effect of glycerol upon these partial pressures.

THE COMPOSITION OF MILK IN BRITISH EAST AFRICA.

By VINCENT H. KIRKHAM, F.I.C., AND A. C. BARNES, A.I.C.

No figures have hitherto been published, except in official reports, concerning the variations in composition of milk in tropical British East Africa. Variations in composition are seasonal, and it is therefore of interest to compare the variations found to occur in the Southern with those occurring in the Northern Hemisphere.

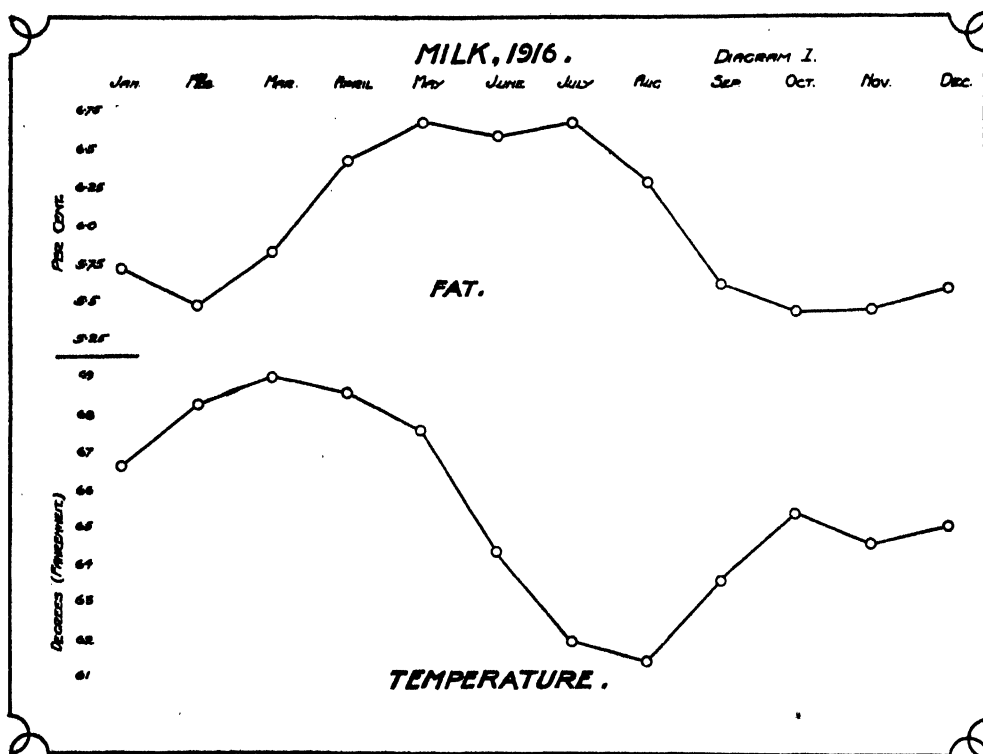


DIAGRAM I.

In 1913 the variations of fat and solids-not-fat during the twelve months were found to show a marked relationship to the variations recorded by Droop Richmond for samples in England, the relationship being that every variation took place in the precisely opposite direction every month—*i.e.*, the graphical representations of the monthly variations of fat and solids-not-fat were in England the mirror images of the East African graphs (Nairobi Laboratory Report, 1913).

In 1916 a more detailed investigation into the composition of milk was carried out, and the results are shown in the accompanying diagrams.

1. FAT CONTENT.

The variation in fat content follows closely the order discovered in 1913. The reason for the variations according to season appears somewhat obscure. Diagram I. gives a representation of the fat variations compared with the mean temperatures each month. No relationship is apparent. Diagram II. gives the same representation of the fat variations compared with the mean temperature variations three

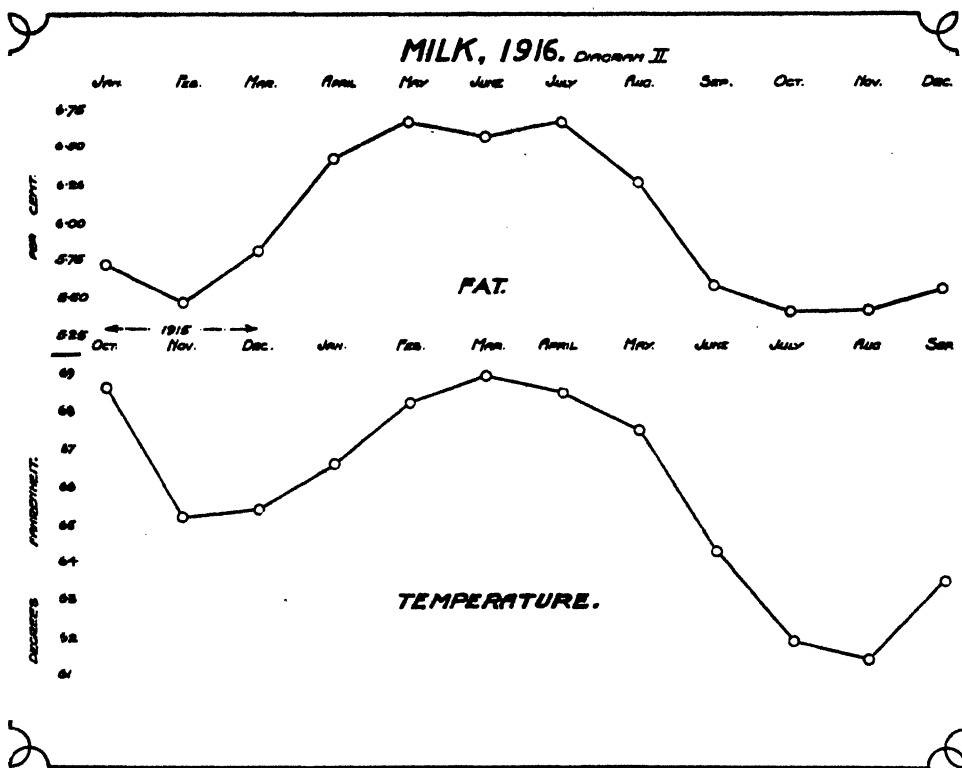


DIAGRAM II.

months previously. A most striking relationship is apparent. If this curious relationship is really of the nature of cause and effect, we have to find an explanation for the time interval of about three months between cause and effect.

At first sight it would seem that the explanation was either (1) that the temperature affects the herbage first, and the altered composition of the grass subsequently alters the composition of the milk; or (2) that the temperature directly affects the cow, but that it takes at least three months for the altered metabolism of the animal to become apparent in the composition of the milk.

Further consideration has led us to correlate this temperature variation with the sexual function of the cow. The period of gestation is nine months, and should the temperature be responsible for the coming in season of the cows, then an explanation

is found for the three months' interval between the "cause" and "effect" upon the milk. We are not in possession of the data respecting calving in the herds from which these samples were taken, but usually there is a rush of calving in September, and consequently the cows are mostly in season during November and December—that is, three parts of the way up the temperature rise. In England cows come into season in early summer—i.e., three parts of the way up the temperature rise—and this explanation therefore serves for both problems—the reversal in point of time in the two hemispheres, and the three months' interval between temperature and fat

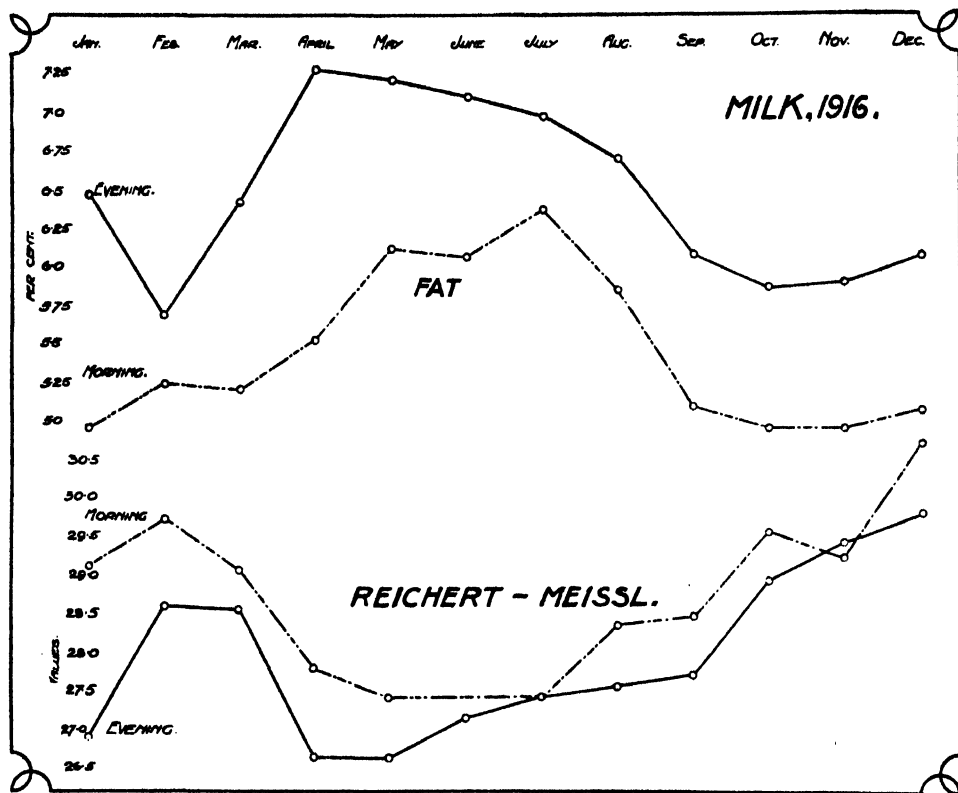


DIAGRAM III.

maxima—the variation in the composition of milk during the lactation period being too well known to require detailing here.

2. COMPOSITION OF THE FAT.

The daily samples of milk were set for cream, and butter was made weekly. From the butters so obtained the Reichert-Meissl figures in Diagram III. were found. It will be seen that there is a marked relationship between the percentage of fat in a milk and the composition of the fat. This, again, is correlated with lactation period.

3. PROTEINS.

This is a very constant figure, and therefore one which is of considerable importance when matters of adulteration are being considered. The comparatively small variations which occur are somewhat erratic, and are not easily correlated with any other observations.

4. MILK SUGAR.

The variation in milk sugar content appears to be influenced by rainfall, though the relationship is not very exact and other influences are evidently at work.

5. MONTHLY AVERAGES.

Month.	Percentages.		
	Fat.	Solids-not-Fat.	Total Solids.
January	5.71	9.12	14.83
February	5.46	9.25	14.71
March	6.42	8.99	15.41
April	6.66	9.24	15.90
May	6.57	9.21	15.78
June	6.66	9.45	16.11
July	6.27	9.68	15.95
August	5.58	9.63	15.21
September	5.41	9.43	14.84
October	5.42	9.24	14.66
November	5.56	9.19	14.75
December	5.96	9.32	15.28

The annual average for this herd of sixty odd cows is above the average for the country, which is:

Fat	...	...	...	...	5.25 per cent.
Solids-not-fat	...	...	...	...	9.25 "
Total solids	...	...	...	...	14.50 "

The cows are mostly native stock (the humped zebu), but grading is being carried out extensively in the country, and the effect of this upon the composition of the milk is not yet ascertained, though, as the quantity is undoubtedly increased, it is to be expected that the quality will deteriorate. Our analyses of milk from grade cows are not sufficient to enable us to state whether this is the case, but investigations into the merits of grade stock are proceeding.

In East Africa the cows are grazing all the year round, and have no cake or corn.

NOTE ON THE REFRACTIVE INDICES OF MIXTURES OF ISOPROPYL ALCOHOL AND ACETONE.

By DOROTHY MURIEL PALMER.

DURING an investigation of the reduction of acetone to isopropyl alcohol it was found that none of the standard chemical methods was of use for the quantitative estimation of small quantities of alcohol in the presence of excess of acetone. An examination of the refractive indices of mixtures of the two substances showed, however, that the relationship between the percentage compositions and refractive indices of the mixtures could be represented by a very flat curve, and it was thought that this might serve as a rapid method for determining the composition.

The chief practical difficulty was due to the extreme volatility of acetone. Little adjustment of the refractometer could be made after placing the liquid in the observation cell, and the time allowed for its temperature to become constant had necessarily to be short.

The acetone was dried over calcium chloride, fractionated, purified by means of its compound with sodium iodide, according to the method of E. A. Werner and Shipsey (*J. Chem. Soc.*, 1913, 103, 1255), and then refractionated under a Young dephlegmator stillhead. The final fraction boiled at 56.45° to 56.55° C. (barometer = 76.63 cm.).

The isopropyl alcohol was prepared from this acetone by reduction with hydrogen in the presence of heated nickel, according to the method of Sabatier. It was then dried over fused sodium sulphate and fractionated three times with an eight-pear column. The final fraction boiled at 80° to 81° C. (barometer, 75.02 cm.).

The readings were taken with a Pulfrich refractometer at a constant temperature of 22° C., values being observed for the hydrogen C and F lines.

Composition. Per Cent. Alcohol.	Refractive Indices.	
	Hydrogen C.	Hydrogen F.
0.0	1.35633	1.36296
1.38	1.35655	1.36324
1.95	1.35651	1.36316
2.41	1.35660	1.36324
3.5	1.35701	1.36358
5.07	1.35714	1.36378
8.24	1.35754	1.36425
9.71	1.35772	1.36436
13.49	1.35834	1.36492
15.1	1.35862	1.36526
16.2	1.35871	1.36534
29.3	1.36097	1.36768
53.8	1.36537	—
67.3	1.36779	1.37443
67.3	1.36763	1.37416
72.3	1.36940	1.37592
89.9	1.37225	1.37861
100.0	1.37470	1.38121

NOTE.

The Editor desires to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

ANALYSIS of drugs having powerful physiological effects is in many cases extremely difficult, particularly in the case of drugs extracted from natural sources, and which are mostly of extremely complex chemical constitution. Great success has recently attended the application of the spectrograph to such problems, the absorption spectra being in many cases in the highest degree characteristic of the substances. For instance, such a minute quantity of pyridine as 0.00001 grm. in 100 c.c. of aqueous ammonia can be readily detected. Much less than 0.5 mgrm. of strychnine, and as little as 3 mgrms. of cocaine have been verified. Very minute quantities of benzoic acid can also be identified by this method—a matter of particular interest, owing to the difficulty of detecting this acid. Again, 1 mgrm. of phenol is shown by its absorption spectrum. A physical method which does not destroy or alter the specimen should be of service to the analyst who has to deal with very small quantities.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Chemical Composition of Cotton-Seed Oil. G. S. Jamieson and W. F. Baughman. (*J. Amer. Chem. Soc.*, 1920, **42**, 1197-1204.)—A sample of oil expressed from Sea Island cotton-seed contained 23 per cent. of saturated fatty acids and 72.5 per cent. of unsaturated fatty acids when separated by the lead-ether method. The unsaturated acids were examined by converting them into bromine addition compounds, and the saturated acids by fractional distillation of their methyl esters, and fractional crystallisation of the fatty acids isolated from the fractions. From the combined results the conclusion was drawn that this cotton-seed oil consisted of glycerides of the following acids: Myristic, 0.3; palmitic, 20; stearic, 2; arachidic, 0.6; oleic, 35.2; and linolic, 41.7 per cent. C. A. M.

Estimation of Dry Solids-not-Fat in Separated Milk. R. Eichhoff. (*Milchwirtsch. Zentralbl.*, 1919, [17]; *Chem. Zeit. Uebersicht*, 1920, **44**, 169.)—The fat-free dry substance in milk, according to Fleischmann's formula, corresponds to $r = 2.665.100 \left(\frac{s-1}{s} \right) + 0.2 f$. If in this formula the weight of unit volume is replaced by the volume of unit weight—i.e., $s = \frac{1}{r}$ —then $r = 2.665.100 (1-r) + 0.2 f$. Now, $1-r$ is the reduction in volume with the increase in the amount of r . If V represents the per cent. by volume, $r = 2.665 - (100 - V) + 0.2 f$. In the

areometer described the values for the diminution in volume are multiplied by 2.665, and the readings give the percentage of dry solids-not-fat in completely separated (fat-free) milk. The results agree closely with those obtained by the usual methods.

C. A. M.

Analysis of Saccharin. O. Beyer. (*Chem. Zeit.*, 1920, **44**, 437-438.)—Saccharin, when submitted to acid hydrolysis (for this purpose the substance may be heated to 150° C. in a sealed capillary tube with a trace of sulphuric acid), yields ammonia and benzene sulphonic acid; the former may be identified by means of Nessler's reagent, and the latter by the test described by Klostermann and Scholta (*ANALYST*, 1916, **41**, 309). *p*-Sulphaminobenzoic acid does not yield these reactions. Saccharin may be estimated in commercial saccharin containing the para-acid (but no other impurity) by titrating 1 grm. of the sample with $\frac{N}{10}$ potassium hydroxide solution, using phenolphthalein as indicator; the percentage of saccharin (x) is calculated by the formula $x = \frac{2.01829 \times c - 100}{0.09845}$, where c is the number of c.c. of $\frac{N}{10}$ alkali solution used.

W. P. S.

Analysis of Wines. W. Fresenius and L. Grünhut. (*Zeitsch. anal. Chem.*, 1920, **59**, 49-79.)—Directions are given in detail for the determination of the sp. gr. of wine by the pycnometer, and when the determination has been made at 17°/4° C., tables are given for correcting the results to 15°/4° C. To estimate the alcohol, the contents of the pycnometer are transferred to a flask and distilled; the sp. gr. of the distillate is determined, and reference to tables gives the corresponding alcoholic strength. Provided that the volatile acidity of the wine is not more than 1.2 grm. per litre (calculated as acetic acid), there is no need to apply a correction or to neutralise the distillate and redistil it. The sp. gr. of the distillation residue, after this has been diluted to its original volume, is also determined, and a table is given showing the weight of total solids per litre corresponding with different sp. grs.

W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Blood Sugar Concentration and Blood Sugar Methods. H. F. Höst and R. Hatlehof. (*J. Biol. Chem.*, 1920, **42**, 347-358.)—This paper gives the results of sugar estimations made on normal and pathological blood and blood to which known amounts of dextrose had been added. On all samples four estimations were made by the following methods—viz: Bang's (*Biochem. Zeitsch.*, 1918, **87**, 264), Hagedorn's (*Ugesk. Læger*, 1918, **80**, 1217), Myers and Bailey's modification of Benedict's (*J. Biol. Chem.*, 1916, **24**, 147), and that of Folin and Wu (*ANALYST*, 1920, **45**, 227).

The results of these comparative estimations indicate that in the case of blood, substances other than dextrose affect the results obtained by each method and to a different extent, so that corresponding results are not in agreement. The highest results are given by the colorimetric methods of Benedict and Folin, while those of Bang and Hagedorn give approximately identical values.

All four methods yield satisfactory results for the dextrose added to the blood, whether normal or pathological.

Tables are provided giving the results obtained by the four methods, and a shorter one showing the average and greatest errors obtained by each method.

T. J. W.

Proximate Analysis of Coniferous Woods. W. H. Dore. (*J. Ind. and Eng. Chem.*, 1920, 12, 476-478.)—*Water*: Two grms. of the fine sawdust are dried at 100° C. *Benzene Extract*: Two grms. dried as above are extracted for six hours, the solvent evaporated, and the residue dried for one hour at 100° C. and weighed. *Alcohol Extract*: The residue from the benzene extraction is extracted for six hours with 95 per cent. alcohol, the solvent evaporated, and the residue weighed. *Cellulose*: The residue from the alcoholic extraction is transferred to a Gooch crucible containing a cloth filtering disc and washed with water, and the cellulose is estimated by the method of Sieber and Walter (*Chem. Absts.*, 1914, 8, 1202). *Lignin*: Two grms. of the material after extraction with benzene and alcohol are dried at about 60° C., and treated for three and a half hours with 20 c.c. of 72 per cent. sulphuric acid. This is treated first with 50 c.c. of cold water and then with 500 c.c. of hot water, and the residue collected in a tared Gooch crucible, washed thoroughly with hot water, dried, and weighed. *Hemicelluloses*: Except in the case of the soluble pentosans, the chlorination washings are unsuitable for the estimation of celluloses, and the presence of lignin derivatives interferes with the reducing sugar methods, or with separate estimations of mannan and galactan. *Soluble Pentosans*: The chlorination filtrates and washings from four cellulose estimations (= 8 grms.) are evaporated to less than 500 c.c., and then made up to that volume, and aliquot portions of 125 c.c. are used for the distillation of furfural. This is precipitated as phloroglucide, dried, and weighed, and the results calculated into xylan by Krob's tables. *Mannan*: Schorger's method of estimation (*ANALYST*, 1917, 42, 362) is used. *Galactan*: Five grms. of the material are mixed with 60 c.c. of nitric acid (sp. gr. 1.15) and the acid evaporated to about 20 c.c. at a temperature not exceeding 87° C. The liquid is diluted with hot water to about 75 c.c. and filtered, and the residual cellulose washed until the filtrate is colourless. The filtrate and washings are evaporated at 87° C. to about 10 c.c., and this residue is set aside for several days for the mucic acid to crystallise, the liquid being finally diluted with 20 c.c. of cold water. The crystals are washed with 20 c.c. of cold water (to dissolve other crystals, possibly of oxalic acid), allowed to stand for twenty-four hours, collected on a tared Gooch crucible containing asbestos, washed successively with 50 c.c. of water, 60 c.c. of alcohol, and finally with ether, dried at 100° C. for three hours, and weighed. The weight multiplied by 1.2 gives the amount of galactan.

C. A. M.

Properties of an Enzymic Extract (Polyzyme) as compared with Malt Diastase. J. Takamine, jun., and K. Oshima. (*J. Amer. Chem. Soc.*, 1920, 42, 1261-1265.)—An aqueous extract of diastatic enzymes, containing several other enzymes, has been prepared by cultivating *Aspergillus oryzae* on media consisting mainly of wheat bran, and is termed "polyzyme." The diastatic power of this preparation does not

decrease at temperatures below 40° C., and the enzymic activity remains unimpaired for more than six months when kept below that temperature. Starch is most readily liquefied by polyzyme in neutral or slightly acid solution, the optimum temperature being 50° C. for a digestion of thirty minutes to two hours, and 40° C. for twenty-four hours' digestion. The preparation is three to five times stronger than ordinary malt extract in its amylolytic power, as tested by Wohlgemuth's method, but has a weaker saccharifying power, as shown by Lintner's method. C. A. M.

Estimation of Hydrocyanic Acid from Beans. A. Czapski. (*Zeitsch. anal. Chem.*, 1920, **59**, 80.)—Paraffin wax is sometimes added to the contents of the distillation flask in the distillation of hydrocyanic acid from cyanogenetic beans in order to prevent excessive frothing. The author finds that the wax hinders the distillation of the acid, and less than one-half of the quantity actually present is found in the distillate. W. P. S.

Estimation of Manganese in Biological Material. C. K. Reiman and A. S. Minot. (*J. Biol. Chem.*, 1920, **42**, 329-345.)—The tissue or blood is placed in a quartz beaker and heated on a sand bath until thoroughly dry. The beaker is then placed over a burner in such a manner that the mouth is most strongly heated, until all tarry matter is destroyed, and is finally placed in an electric furnace at 600° to 700° C. until nearly all the carbon has disappeared.

To the residue about 3 grms. of a mixture of sodium and potassium nitrates is added, followed by 10 c.c. of sulphuric acid and 5 c.c. of hydrochloric acid, when the beaker is gradually raised in temperature to a cherry-red heat. If a large amount of carbon be left, a few c.c. of hydrochloric, nitric, and sulphuric acid should be added, and the fusion repeated. When cooling, the beaker should be rotated in order to prevent the fusion solidifying into a large cake. Fifty c.c. of water are added, and the beaker heated upon a sand bath until the contents have dissolved, when the solution is filtered. To the solution 1 c.c. of concentrated nitric acid is added, about 0.3 c.c. of 2.5 per cent. silver nitrate solution, and about 0.5 gm. of potassium persulphate; the mixture is then gently warmed for ten minutes.

A standard solution of potassium permanganate is prepared, completely reduced by the addition of sulphurous acid, and portions of known volume treated with nitric acid, silver nitrate, and potassium persulphate as above; finally, the pink colour is developed by gentle heating. In this manner a series of Nessler tubes containing from 0.002 to 0.025 mgrm. of manganese is prepared, and used for comparison with the solution to be estimated.

The method is a modification of the one devised by Bertrand (*Compt. rend.*, 1905, **141**, 1255, etc.), and yields more accurate results than the original method. The authors had much difficulty in obtaining apparatus free from manganese, and recommend that the beakers, etc., employed should be frequently submitted to blank tests during the course of the work. Tables are furnished comparing the results obtained by the present method and Bertrand's original method, the manganese content of normal and pathological human blood, and of various human viscera.

T. J. W.

Method for the Determination of Methæmoglobin and Hæmoglobin in Blood. W. S. McElroy. (*J. Biol. Chem.*, 1920, **42**, 297-300.)—One c.c. of oxalated blood is transferred to a 50 c.c. flask containing 20 c.c. of distilled water. One c.c. of 4 per cent. potassium ferricyanide solution is added, and the flask is shaken to insure thorough mixing. After standing for five minutes the solution is diluted to 50 c.c., mixed, and the colour compared with a standard solution of methæmoglobin 10 or 15 mm. in length in a colorimeter. The result gives the total hæmoglobin (hæmoglobin + methæmoglobin) in grms. per 100 c.c. of blood. A further portion of the same blood is used to determine the oxygen capacity by the Van Slyke method (*J. Biol. Chem.*, 1918, **33**, 127), and from this the hæmoglobin content is calculated in grms. per 100 c.c.

The difference between the "total hæmoglobin" and the hæmoglobin gives the methæmoglobin content of the blood.

The standard solution of methæmoglobin is prepared from normal oxalated ox blood by the above method, using ten times the volumes given, and the hæmoglobin content is calculated from the oxygen capacity experimentally obtained. Should the standard be darker in colour than the solution under examination, it is diluted until the colours are approximately of the same intensity before comparison in the colorimeter. The method gives results rapidly and accurately.

T. J. W.

Detection of Urea. J. F. A. Pool. (*Pharm. Weekblad*, 1920, **20**, 178; *Chem. Zeit. Uebersicht.*, 1920, **44**, 165.)—When microscopic sections of soya beans are placed on a gelatin plate containing about 2 per cent. of urea and an aqueous extract of yeast, the urease in the beans decomposes the urea into ammonium carbonate, the calcium carbonate and calcium phosphate of the yeast are precipitated in amorphous condition, and colorations are produced on the surface of the plate (Beijerinck). In the absence of yeast, a plate containing 2 per cent. of agar-agar, 2 per cent. of urea, and a little phenolphthaleïn may be used for the detection of urease. Conversely, urea may be detected by this means in water, urine, etc. A small quantity of the liquid under examination is rendered neutral to phenolphthaleïn and boiled with 2 per cent. of agar-agar. The mass is poured on to a clock glass, a section of a soya bean applied to it after solidification, and the glass covered. In the presence of urea ammonia is liberated, and a red coloration spreads over the surface of the bean. In this way it is possible to detect 1 mgrm. of urea in 1 c.c. of liquid.

C. A. M.

Determination of Vitamine. R. J. Williams. (*J. Biol. Chem.*, 1920, **42**, 259-265.)—The author has previously shown that the anti-beri-beri vitamine is necessary for the nutrition of yeast (*J. Biol. Chem.*, 1919, **38**, 465), and this fact has been adapted to the estimation of the amount of vitamine present in solution. The rate of growth of yeast and the number of cells produced from one cell under standard conditions in eighteen hours is directly proportional to the amount of vitamine present.

This microscopical method, although yielding good results, is troublesome, and

the following gravimetric method, which yields more accurate results, has been adopted: The culture solution is of the usual type, containing cane sugar, asparagine, and various salts in water; 100 c.c. of this solution are placed in a 500 c.c. flask, the solution to be tested added, and the whole diluted with water to 110 c.c. The flask is plugged with cotton-wool, sterilised, and cooled to 30° C.

A suspension of fresh pressed yeast is made containing 0.3 grm. in 1 litre of sterile water, well shaken, and 1 c.c. is introduced into the culture medium with a sterile pipette. The flask is placed in an incubator at 30° C. for eighteen hours, and the growth is then stopped by the addition of a little formalin. The yeast is filtered off through a weighed Gooch crucible, washed with water and alcohol, dried at 103° C., and weighed. The increase in weight over that of a blank determination is directly proportional to the vitamine solution added. The "vitamine number" of a material is defined as the number of mgrms. of yeast produced by the addition of its extract computed to 1 grm. of the original material tested.

A discussion of the method is given, in which it is shown that the vitamine to be tested must be in solution, that foreign organisms present in the yeast offer no serious handicap to the working of the method, and that the usual substances present in vitamine solutions do not accelerate the growth of the yeast. T. J. W.

ORGANIC ANALYSIS.

New Method for the Estimation of Acetic Acid in Acetates. O. A. Pickett. (*J. Ind. and Eng. Chem.*, 1920, **12**, 370-371.)—The acetate is distilled with xylene in place of the water used in the usual method; 2.5 grms. of the acetate are mixed with 30 c.c. of water, 20 c.c. of 85 per cent. phosphoric acid, and 350 c.c. of xylene, and the mixture is distilled until only a thin film of xylene remains on the surface of the phosphoric acid solution in the distillation flask. The total distillate is then titrated. If the sample contains chlorides, allowance must be made for the hydrochloric acid in the distillate. W. P. S.

Modification of the Van Slyke Method for determining Arginine. A. E. Koehler. (*J. Biol. Chem.*, 1920, **42**, 267-268.)—Previous work by various experimentalists (*Am. J. Physiol.*, 1908-09, **23**, 194) has shown that arginine liberates one-half of its nitrogen as ammonia when boiled with concentrated alkali, and the apparatus described was devised to overcome the bumping produced when boiling such a solution. The apparatus consists of a flask for containing the boiling solution fitted with a long vertical reflux condenser and an inlet tube so arranged that a current of air may be drawn through the liquid after first passing through a wash bottle. To the top of the condenser is fitted an exit tube connected to a wash bottle containing the standard acid for absorbing the evolved ammonia, and the exit tube from this wash bottle is connected to a filter pump.

A slow current of air is passed through the apparatus during the heating and boiling of the solution, and this serves to prevent bumping and also to convey the

liberated ammonia into the standard acid. By using $\frac{N}{14}$ acid the value in c.c. neutralised is equal to the mgrms. of nitrogen in the ammonia absorbed.

T. J. W.

Comparative Method of determining the Heat of Carbonisation of Coal. G. Weyman. (*J. Soc. Chem. Ind.*, 1920, 39, 168-169t.)—It is found in practice that the rate at which coal can be passed through continuous vertical retorts with efficient coking and with the same supply of heat to the heating flues varies up to 30 per cent. according to the type of coal used. To determine this variation experimentally, the author used a fireclay furnace, 8 inches in external diameter, enclosed in a metal vessel, from which it was separated at the sides and bottom by asbestos packing. A crucible containing 1 kgrm. of copper was placed in the furnace, and the metal melted by a blowpipe operating through an aperture in the vessels and lining; when the copper was molten the blowpipe was withdrawn, the hole packed with asbestos, and the asbestos lid placed in position. As soon as the first crystals of copper appeared, or when the temperature of the copper fell to some predetermined temperature (as read by a pyrometer), a thin steel tube containing a weighed quantity of the powdered coal was thrust into the middle of the copper. The tube communicated with a wash bottle and with a meter which was read every minute until the evolution of gas had practically ceased. The tube was then removed, and the pellet of coke examined. Experiments with different coals showed that the method yielded consistent results and gave a true indication of the comparative amounts of heat required to carbonise the coals.

W. P. S.

Analysis and Composition of Cresylic Acid. J. J. Fox and M. F. Barker. (*J. Soc. Chem. Ind.*, 1920, 39, 169-172t.)—The phenol present may be estimated by a process described previously by the authors (*ANALYST*, 1917, 42, 329), whilst Raschig's method (*ibid.*, 1900, 25, 298) is trustworthy for the estimation of *m*-cresol. Another method for estimating *m*-cresol consists in treating about 1 grm. of the mixed cresols with a solution of bromine in carbon tetrachloride, removing the solvent at a temperature below 50° C. under reduced pressure, and determining the increase in weight due to bromination. *m*-Cresol yields a tribromo derivative, whilst phenol, *o*-cresol, and *p*-cresol yield dibromo derivatives. If *W* is the weight of bromo derivative obtained from 8 grms. of the mixed cresols, the percentage of *m*-cresol is $(100W - 246.3)/0.731$. A modification of Ditz and Cedivoda's method (*ibid.*, 1900, 25, 74) may also be used for the purpose. About 1 grm. of the cresol is mixed with 5 c.c. of 2*N* sodium hydroxide solution and diluted to 200 c.c.; 10 c.c. of this solution are then treated with an excess (100 c.c.) of a mixture of equal parts of $\frac{N}{20}$ potassium bromide solution and $\frac{N}{160}$ potassium bromate solution, and 10 c.c. of hydrochloric acid. After the addition of 20 c.c. of 10 per cent. potassium iodide solution (this is added under conditions which prevent loss of bromine vapour) the mixture is placed aside for one hour and the excess of iodine then titrated. One grm. of *m*-cresol absorbs 4.444 grms. of bromine, whilst 1 grm. of *o*-cresol or *p*-cresol absorbs 3.080 grms. If *Br* is the actual weight of bromine absorbed, the percentage of *m*-cresol in a mixture is

(100Br - 308)/1.364. The data obtained in the above three methods in conjunction with the sp. gr. afford the means for determining the *o*-cresol and *p*-cresol in a mixture of cresols. The sp. gr. at 15.5°/15.5° C. of phenol is 1.0774, of *o*-cresol 1.0516, and of *m*-cresol and *p*-cresol 1.0388. The formula connecting sp. gr. with composition is :

$$\text{Sp. gr.} = [1.0774 \times P + 1.0388 \times M + 1.0516 \times O + 1.0388 (100 - P - M - O)]/100,$$

where *P*, *M*, *O*, and (100 - *P* - *M* - *O*) are the percentages of phenol, *m*-cresol, *o*-cresol, and *p*-cresol respectively. This expression simplifies to $0.0386P + 0.0128O = 100$ (sp. gr. - 1.0388), in which *O* is the only unknown quantity. If phenol is not present, the equation is simply $0.9128O = 100$ (sp. gr. - 1.0388). Approximate results for *o*-cresol and *p*-cresol may be calculated from the refractive index at 50° C. of a mixture of cresols ; the refractive index of *o*-cresol is 1.5309, of *m*-cresol 1.5266, and of *p*-cresol 1.5260. The percentage of *o*-cresol (*O*) is given by the formula—

$$O = (100n_D - 0.0006M - 152.6)/0.0059,$$

where *M* is the percentage of *m*-cresol and *n_D* the observed refractive index at 50° C. Analyses of cresylic acids are recorded showing that the proportions of the different constituents vary considerably in different makes. W. P. S.

Colour Changes of the Diphenylamine Reaction. E. M. Harvey. (*J. Amer. Chem. Soc.*, 1920, **42**, 1245-1247.)—Three distinct colorations may be produced in the diphenylamine reaction for nitric nitrogen, the nature and intensity of these colorations varying with the concentration of the sulphuric acid. In testing plant tissues for nitric nitrogen the best coloration is obtained when the concentration of the sulphuric acid is kept at about 72 per cent. It is also necessary to avoid variations of temperature in making a series of tests. For micro-chemical purposes the following modified reagent has the advantage that additional moisture will not affect the optimum conditions for the maximum coloration : Diphenylamine, 0.05 gm. ; 95 to 96 per cent. sulphuric acid, 7.5 c.c. ; and 10 per cent. potassium chloride solution, 2.5 c.c. C. A. M.

Analysis of Hydrogenated Oils. A. Grün. (*Chem. Umschau.*, 1920, **26**, 101 ; *Chem. Zeit. Uebersicht.*, 1920, **44**, 171.)—A trustworthy method of distinguishing between hydrogenated marine animal oils and hydrogenated rape oil has been based upon the determination of the molecular weights of the fatty acids of lower b.-pt. fractionated under reduced pressure. Only the hydrogenated marine animal oils contain acids with molecular weight lower than that of palmitic acid, whilst hydrogenated rape oil yields no acid fraction with molecular weight exceeding 201. In the presence of a fat of the coconut oil group, which may be recognised by the higher proportion of caprylic and lauric acid, the above method is untrustworthy. Hydrogenated marine animal and rape oils may be distinguished from other hydrogenated oils by containing a larger proportion of behenic acid or of other acids with higher molecular weight than stearic acid. In this case the least soluble fatty acids are separated by fractional crystallisation from various solvents, and their molecular equivalent determined. C. A. M.

Critical Study of Methods for the Detection of Methyl Alcohol. A. O. Gettler. (*J. Biol. Chem.*, 1920, 42, 311-328.)—The author has examined fifty-eight tests described in the literature by employing them with the distillates from a number of liquors to which known amounts of methyl alcohol were added ranging from nil to 30 per cent., in addition to five typical confiscated methyl alcohol liquors—a total of eighteen. The various reactions are divided into two groups: (A) Those in which the methyl alcohol must be oxidised to formaldehyde before testing, and (B) those in which the methyl alcohol is tested for directly.

On the whole, the former class yields more reliable and sensitive reactions, and is subdivided according to the class of compound with which the formaldehyde reacts—*e.g.*, phenylhydrazines, phenols, alkaloids, proteins, amines, and miscellaneous substances.

The most satisfactory and sensitive reactions are: (a) Phenylhydrazine-ferric chloride-hydrochloric acid, (b) phenylhydrazine-sodium nitroprusside-sodium hydroxide, (c) apomorphine-sulphuric acid, (d) peptone-ferric chloride, (e) reduced fuchsine-sulphuric acid and two crystal-producing tests, (f) β -naphthol-hydrochloric acid, and (g) hexamethylenetetramine-mercuric chloride.

The five colour reactions are sensitive to 1 in 200,000, but the crystal-forming reactions are reliable down to 5 per cent. of methyl alcohol, and in solutions containing less than this amount the alcohol must be concentrated by fractional distillation.

Of the reactions classed under the heading of Group (B), only two out of twelve appear to be reliable and easily performed. These are—(h) boiling for seven hours with hydroxylamine and potassium hydroxide under a reflux condenser, when cyanide is produced and may be tested for in the usual way, and (i) determination of the sp. gr. and refractive index of the solution. The former test is very sensitive, while the latter is reliable for not less than 5 per cent. of methyl alcohol, and is liable to interference by the presence of substances other than ethyl and methyl alcohols and water.

Detection of methyl alcohol in liquors: 100 c.c. of the liquor are neutralised with sodium carbonate to phenolphthalein, and slowly distilled until 50 c.c. of distillate are collected. This is divided into two portions of 30 c.c. and 20 c.c., the latter being tested directly by the above reactions (h) and (i). To the 30 c.c. portion of distillate 100 c.c. of 10 per cent. sulphuric acid are added, followed by 6 grms. of potassium dichromate, and the whole allowed to stand ten minutes. The flask is connected to a condenser and distilled, so that about 30 c.c. of distillate are collected in one hour. This distillate contains most of the acetaldehyde and is rejected, and distillation then continued somewhat more rapidly until about 60 c.c. are obtained. This fraction, which contains nearly all the formaldehyde, is then tested by the above reactions in the following sequence: (b), (c), (d), (e), (g), (f). A definite reaction with one or two of these tests only may be due to foreign substances, but if methyl alcohol was originally present, all the results obtained will be positive.

Detection of methyl alcohol in tissues: 500 grms. of tissue are finely ground and placed in a large distillation flask with 500 c.c. of water. Sulphuric acid is added until a distinctly acid reaction is produced, and the mixture is then distilled in a current of steam. Three hundred c.c. are collected, neutralised, and again distilled

slowly, this distillate being oxidised as above, when subsequent distillation gives all the formaldehyde in the first 40 c.c. The final distillate is then tested by the same reagents as given above under examination of liquors, omitting the hexamethylene-tetramine mercuric chloride test.

Full details are given for the preparation and employment of the various reagents, which are classed according to their reliability, sensitiveness, and ease of application.

T. J. W.

Estimation of Nitro Groups by Young and Swain's Method. L. Desvergues. (*Ann. Chim. anal.*, 1920, 2, 141-143.)—This method (*ANALYST*, 1897, 22, 329), in which the nitro group is reduced with stannous chloride and excess of the latter titrated with iodine solution, yields trustworthy results with many organic nitro compounds; in the case of nitrophenols, nitrocresols, and nitronaphthalenes, however, the results obtained are too low. The author uses more alcohol (50 c.c.) than is prescribed by Young and Swain for dissolving the substance previous to the reduction.

W. P. S.

Diazometric Estimation of Phenol and of Certain of its Homologues. R. M. Chapin. (*J. Ind. and Eng. Chem.*, 1920, 12, 568-570.)—The method depends on the quantitative coupling of phenols (phenol, cresols, xylenols) with diazonium salts to form insoluble hydroxyazo compounds. The reagent used is prepared by dissolving 14 grms. of *p*-nitraniline in 400 c.c. of water and 70 c.c. of concentrated nitric acid, heating the solution on a water-bath for two hours, allowing it to cool overnight, then filtering and diluting the filtrate to 1 litre; a portion of this solution is mixed with an equal volume of 1 per cent. sodium nitrate solution five minutes before it is required for a titration. This diazo solution may be standardised against β -naphthol. For the estimation, 20 c.c. of an approximately $\frac{N}{10}$ phenol solution are mixed with 50 c.c. of 10 per cent. sodium acetate solution, the mixture is neutralised with acetic acid, 10 c.c. of 30 per cent. basic lead acetate solution are added, and the diazo solution is run in from a burette with stirring. A further 10 c.c. of basic lead acetate solution is added as each 10 c.c. of the diazo solution is used. When the end of the reaction is near, as shown by spot test with drops of diazo solution and phenol solution respectively, the mixture is left at rest for two minutes, again stirred, and the point of a folded filter is immersed in the mixture. Two small quantities of the liquid passing inwards into the filter are transferred to a test-plate, and one is tested with phenol solution and the other with diazo solution. If no colour develops in either, a drop of 25 per cent. sodium hydroxide solution is added to each. If the end-point is at some distance on either side, the distinction is sharp, but if it is near, a strong coloration may appear in both tests. The titration may be continued until the end-point is clearly passed, and the limits thus coarsely defined are narrowed down by subsequent titrations.

W. P. S.

Durability of Exterior Varnishes compared with their Physical and Chemical Analyses. W. T. Pearce. (*J. Ind. and Eng. Chem.*, 1920, 12, 552-555.)—The physical tests made were viscosity, sp. gr., and time of drying of the

varnish, and the elasticity, brilliancy, and resistance to moisture of the dry film; the working characters of the varnishes under the brush were also noticed, and varnished wooden surfaces were exposed in the open air, observations of their surface, etc., being made at the end of nine and twelve months respectively. The chemical analysis included estimations of the thinner, petroleum products in the thinner, non-volatile matter, and ash, resin, and oil. Comparison of the results and data obtained showed that there is a relationship between the viscosity, surface, elasticity, moisture test, etc., and the service (exposure) test.

W. P. S.

INORGANIC ANALYSIS.

Titration of Ammonium Hydrogen Fluoride. W. S. Chase. (*J. Ind. and Eng. Chem.*, 1920, 12, 567-568.)—Direct titration of ammonium hydrogen fluoride yields untrustworthy results owing to the fact that all the usual indicators are useless in the presence of free hydrofluoric acid. If, however, the hydrofluoric acid is precipitated by the addition of calcium chloride, the equivalent quantity of hydrochloric acid liberated may be titrated, using methyl orange as indicator. About 6 grms. of calcium chloride dissolved in 75 c.c. of water are added to 1.4 grms. of the fluoride dissolved in 125 c.c. of water, the mixture is stirred for one minute, and then titrated without filtration.

W. P. S.

Rapid Method for the Estimation of Arsenic in Commercial Sulphuric Acid. A. A. Kohr. (*J. Ind. and Eng. Chem.*, 1920, 12, 580-581.)—To estimate the arsenious acid present, 20 grms. of the sulphuric acid are diluted with a small quantity of water, methyl orange is added, and the mixture neutralised with saturated sodium carbonate solution, but the indicator must still exhibit a very faint pink coloration; 2 grms. of sodium hydrogen carbonate are then added, the solution diluted to 250 c.c., and titrated with $\frac{N}{10}$ iodine solution. For the estimation of arsenic acid, another portion of 20 grms. of the sulphuric acid is heated at 105° C. for one hour (this will remove nitrous and nitric acids), then diluted and treated with a slight excess of saturated sodium carbonate solution (red coloration with phenolphthalein); the mixture is boiled, filtered, and the filtrate treated with 3 grms. of sodium hydrogen carbonate, 150 c.c. of concentrated hydrochloric acid, and 1 gm. of potassium iodide. After five minutes the liberated iodine is titrated with $\frac{N}{10}$ thio-sulphate solution; each c.c. of the latter is equivalent to 0.00495 gm. of As_2O_3 present in the arsenic form. The sum of the results of the two estimations gives the total quantity of arsenic as arsenious acid. Copper interferes with the estimation of the arsenic acid, and if an appreciable quantity of this metal is present it should be estimated and an allowance made for its amount; 1 molecule of copper liberates 1 molecule of iodine from potassium iodide.

W. P. S.

Influence of Atmospheric Oxygen on the Iodimetric Estimation of Chromium. O. Meindl. (*Zeitsch. anal. Chem.*, 1919, 58, 529-547.)—The statement of Wagner that potassium dichromate shows an increased iodimetric activity when subjected to the direct action of hydriodic acid has been confirmed, and has been

shown to be due to "activation" of the oxygen in the primary reaction between the chromic acid and hydriodic acid. The more slowly this reaction proceeds the greater is the induced activity, so that variable results are obtained under different conditions. Hence Zulkowsky's method (*J. prakt. Chem.*, 1868, 103, 351) is unsuitable for the accurate standardisation of iodine solutions. C. A. M.

Estimation of Nitrates and Nitrites in Sulphuric Acid. L. B. Sefton. (*Chem. Trade J.*, 1920, 66, 755-757.)—The diphenylamine test for nitrates is useless for the examination of battery acid, since the characteristic blue coloration is given by nitrites, iron, and arsenates, and by sulphuric acid itself. Denigès's strychnine reagent gives satisfactory results if applied as follows: Five c.c. of a 1 per cent. solution of strychnine sulphate are mixed immediately before use with 5 c.c. of concentrated hydrochloric acid, and reduced by treatment for ten minutes with 4 to 5 grms. of amalgamated zinc. Ten c.c. of the battery acid (sp. gr. about 1.28) are mixed with 0.5 c.c. of the reagent, and then with 10 c.c. of concentrated sulphuric acid (sp. gr. 1.83). In the presence of nitrates or nitrites a pink coloration, which is permanent for several hours, will develop. The reaction is unaffected by 0.005 to 0.01 per cent. of ammonia, arsenic, copper, or chlorides. A solution containing 0.005 per cent. of iron gives an orange coloration which interferes with the test. The method will detect 1 part in 5,000,000 of nitrates or nitrites, and is more stable than the coloration given by brucine (0.2 gm. in 100 c.c. of pure strong sulphuric acid); the latter reaction is not affected by 0.005 to 0.025 per cent. of iron, arsenates, arsenites, chlorides, copper, or ammonia. In solutions of acid of sp. gr. below 1.7, nitrites react to the same extent as nitrates, but nitrites in solutions of sp. gr. 1.83 have no effect on the reagent. The most suitable sp. gr. of acid for applying the test is 1.65 to 1.7, and the speed of the reaction is increased by raising the temperature. The reagent should be freshly prepared for each test. In fresh electrolytes the method is sensitive to 1 part in 5,000,000, and in used electrolytes to 1 part in 400,000. A more sensitive reagent is prepared by dissolving 1 gm. of brucine in 2 c.c. of concentrated sulphuric acid, and mixing the solution with 50 c.c. of water immediately before use. Of the dilute solution, 5 c.c. are mixed with 10 drops of concentrated sulphuric acid, and reduced by treatment for five to ten minutes with 4 or 5 grms. of amalgamated zinc. Two c.c. of the resulting solution are used for each test. For the detection of nitrites the most satisfactory reagent is prepared by dissolving 2 grms. of dimethylaniline in 3 c.c. of strong hydrochloric acid and 25 c.c. of water; 3 drops of the reagent are added to 25 to 30 c.c. of the battery acid, and the mixture allowed to stand for ten to thirty minutes. The yellow coloration produced in the presence of nitrites is compared with standards under similar conditions. The test is capable of detecting 0.0001 per cent. of nitrites in fresh or 0.0004 per cent. in used, battery acid, and is not affected by the presence of 0.005 to 0.1 per cent. of any other impurities. C. A. M.

Short Commercial Analytical Methods for the Determination of Purity of Important Chemicals used in Pyrotechnics. H. B. Faber and W. B. Stoddard. (*J. Ind. and Eng. Chem.*, 1920, 12, 576-578.)—Small amounts of sodium

nitrate in potassium nitrate may be estimated by precipitation as caesium-sodium-bismuth nitrite (*cf.* Ball, *ANALYST*, 1910, 35, 81). To estimate aluminium in aluminium flake or powder, 2 grms. of the sample are mixed in a fireclay crucible with 100 grms. of litharge and 30 grms. of borax, and the mixture is covered with a layer of 25 grms. of borax glass. The mixture is fused for twenty minutes at a bright red heat, cooled, the lead button separated from the slag, and weighed. The weight of the lead multiplied by 0.0872 gives the weight of the aluminium. The quantity of pure nitrate in a sample of commercial nitrate may be estimated by heating 1 gm. of the sample with 5 grms. of ignited tungstic anhydride; oxygen and nitrogen oxides corresponding empirically with N_2O_5 are liberated, and the amount of nitrate may be calculated from the loss in weight.

W. P. S.

Sensitiveness of Qualitative Reactions. I. Potassium. O. Lutz. (*Zeitsch. anal. Chem.*, 1920, 59, 145-165.)—With the object of obtaining a uniform view of the sensitiveness of various reagents for potassium, the author suggests that the tests should be made as far as possible under the same conditions. The following are the results found by treating 5 c.c. of potassium chloride solution with the various reagents, the temperature being 18° C. in each case, and the observation made after five minutes' contact; the figures represent the lowest concentration at which potassium can be detected: Perchloric acid, 1 : 435; phosphomolybdic acid, 1 : 561; platinum chloride, 1 : 587; sodium borofluoride, 1 : 970; aniline hydrosilicofluoride, 1 : 1,022; sodium tungstate, 1 : 2,170; phosphotungstic acid, 1 : 2,809; sodium cobalt nitrite, 1 : 25,000; sodium bismuth thiosulphate, 1 : 57,000; sodium picrate, 1 : 840; sodium-naphthol sulphonate, 1 : 1,022; sodium hydrogen tartrate, 1 : 1,050. It is also suggested that the results might be expressed in actual weights of potassium; for instance, perchloric acid will detect 1 part of potassium in 435 parts or 11×10^{-3} gm. of potassium.

W. P. S.

Preparation of Sodium Amalgam in Flakes. A. D. Hirschfelder and M. C. Hart. (*J. Ind. and Eng. Chem.*, 1920, 12, 499.)—A simple method of preparing flocculent sodium amalgam for organic work is to pour the melted amalgam slowly into xylene or kerosine, which is rapidly agitated by an electric stirrer. The flocculent deposit is dried on a porcelain plate in a current of air, and may then be readily pulverised.

C. A. M.

Estimation of Minute Quantities of Oil in Sulphur. L. S. Bushnell and H. S. Clark. (*J. Ind. and Eng. Chem.*, 1920, 12, 485.)—About 50 grms. of the powdered sulphur are shaken in a closed flask with about 50 c.c. of redistilled petroleum spirit at intervals of thirty minutes for several hours, and the extract then decanted through a filter. This treatment is repeated until the whole of the sulphur has been extracted, and the united extracts are then placed in a Wiley extraction apparatus in which has been suspended a roll of sheet copper foil (4 ins. $\times$ 15 ins.), in which no two points of the surface of the metal come in contact. This copper coil is cleaned with dilute nitric acid, washed with water and alcohol, and dried with ether before use. On boiling the petroleum spirit the whole of the dissolved sulphur will

be deposited on the copper, and the solvent is then filtered, the filtrate and washings evaporated, and the residue of oil weighed. C. A. M.

Application of the Rotating Zinc Reductor to the Estimation of Molybdenum. W. Scott. (*J. Ind. and Eng. Chem.*, 1920, 12, 578-580.)—Molybdic acid in dilute sulphuric acid solution is reduced readily by means of a rotating zinc cylinder (*cf.* ANALYST, 1920, 60), and there is no need to use a current of electricity. The temperature of the solution should be between 20° and 30° C., and when the reduction is complete the mixture is titrated in the same vessel with standardised permanganate solution. If desired, the reduced molybdenum solution may be transferred to another vessel containing iron alum solution and phosphoric acid before titration; in this case the presence of the iron alum and phosphoric acid is essential. W. P. S.

Estimation of Zirconium in Steel. G. E. F. Lundell and H. B. Knowles. (*J. Ind. and Eng. Chem.*, 1920, 12, 562-567.)—A method is given for the estimation of silicon, aluminium, titanium, and zirconium in steels which may possibly contain tungsten, chromium, vanadium, phosphorus, molybdenum, copper, nickel, cobalt, uranium, and cerium. Five grms. of the steel are dissolved in hydrochloric acid with the addition of a small quantity of nitric acid, the solution is evaporated to dryness, the residue baked, dissolved in dilute hydrochloric acid (1 : 1), the insoluble silica is collected, ignited, weighed, evaporated with hydrofluoric acid, and any remaining residue dissolved after fusion with potassium pyrosulphate. The hydrochloric acid solution is evaporated to a syrup, taken up with 40 c.c. of hydrochloric acid (1 : 1), and extracted with ether to remove most of the iron and molybdenum. The acid solution is heated to expel dissolved ether, the residue recovered from the silica is added, the solution oxidised with nitric acid, diluted, cooled, and treated with an excess of 20 per cent. sodium hydroxide solution. The precipitate is collected on a filter, dissolved in hydrochloric acid, again precipitated, and collected. (Filters from which precipitates have been dissolved should be ignited, the ash fused with sodium carbonate, washed with water, then dissolved in hydrochloric acid, and the solution added to the main bulk.) *Estimation of Aluminium:* If chromium and uranium are absent, the united filtrates from the sodium hydroxide precipitation are acidified with hydrochloric acid, boiled, and rendered just ammoniacal. Aluminium is precipitated as hydroxide contaminated with phosphate; the precipitate is collected, dissolved in hydrochloric acid, the solution diluted to 50 c.c., rendered ammoniacal, then acidified with nitric acid, heated to 50° C., and the phosphoric acid separated as ammonium phosphomolybdate. After filtration, the alumina is precipitated from the filtrate by ammonia, collected, ignited, and weighed. A trace of silica may be present, and is removed by treating the ignited aluminium oxide with hydrofluoric acid in the usual way. If chromium is present, the solution must be oxidised with bromine before the final precipitation of the aluminium, and, when uranium is present, this final precipitation must be made with ammonium carbonate in place of ammonia. In the case of steels containing vanadium, the aluminium hydroxide, after ignition, must be fused with pyrosulphate, the melt dissolved in dilute sulphuric

acid, the vanadium reduced and titrated with permanganate; the amount of vanadium oxide thus found is deducted from the weight of the aluminium oxide. *Estimation of Zirconium and Titanium*: The sodium hydroxide precipitate (see above) is dissolved in warm dilute hydrochloric acid, the solution diluted to 250 c.c., partially neutralised with ammonia (the solution should still contain 5 per cent. of hydrochloric acid), 2 grms. of tartaric acid are added, and the iron is reduced with hydrogen sulphide. Insoluble sulphides (if formed) are separated by filtration, the filtrate is rendered ammoniacal, and treated further with hydrogen sulphide. The precipitated iron, nickel, cobalt, and manganese sulphides are collected on a filter and washed with dilute ammonium chloride-ammonium sulphide solution. The filtrate is acidified with sulphuric acid, heated on a water-bath to coagulate sulphur, filtered, the filtrate cooled in ice-water, and treated with an excess of 6 per cent. "cupferron" solution. After ten minutes, the precipitate is collected, washed with cold 10 per cent. hydrochloric acid, ignited in a platinum crucible, and the combined zirconium and titanium oxides are weighed. The oxides are then fused with potassium pyrosulphate, the melt dissolved in 10 per cent. sulphuric acid, and the titanium estimated colorimetrically or volumetrically.

W. P. S.

APPARATUS, ETC.

Colorimetric Determinations with Solutions containing two Coloured Substances. K. G. Falk and H. M. Noyes. (*J. Biol. Chem.*, 1920, 42, 109-130.)—This paper gives details of an investigation into the principles underlying the quantitative estimation of dextrose by its action upon sodium picrate, resulting in the formation of sodium picramate. A Duboscq colorimeter was employed throughout, with a Mazda 75-watt electric lamp, and all the usual precautions necessary in colorimetric work were observed. The chief conclusions reached are as follows: The addition of sodium hydroxide to picrate solution deepens the colour, but sodium carbonate has no action unless the concentration is greater than three equivalents of carbonate to one of picric acid.

The colour values of free picramic acid are not proportional to the concentration and exposure to light deepens the colour. Sodium picramate is unaffected by excess of sodium hydroxide or carbonate, and by exposure to electric light for twenty-four hours.

Readings obtained with mixed solutions of sodium picrate and picramate give relative values ranging from 1-200 to 1-1,500, according to the proportion of each salt present, thus causing uncertainty in the results. Solutions of sodium picrate reduced by dextrose yield darker solutions with increased time of heating; the presence of other salts tends to reduce the rate of colour formation. The action of dextrose on sodium picrate solution gives rise to the formation of small amounts of substances other than sodium picramate, which adversely affect the accuracy of the readings.

Accurate results may be obtained by matching picramate solutions against a solution of potassium dichromate, providing that the latter is always of the same concentration and the same depth of solution is employed. The most accurate results are obtainable by matching the colour of a solution against a standard similar in composition and concentration.

T. J. W.

Combination of Fractionation with Photospectrometry in Organic Analysis. W. E. Matthewson. (*J. Amer. Chem. Soc.*, 1920, **42**, 1277-1279.)—A method of estimating colourless substances in quantities of less than a few mgrms. consists in combining or condensing the substance to form a strongly coloured derivative, which is then separated by fractionation with an immiscible solvent from other coloured substances in the mixture, and subjected to spectrophotometric examination. Suitable reagents for the formation of coloured compounds are picryl chloride, dinitrophenyl-hydrazine, and diazobenzene-sulphonic acid. For example, in the case of acetone, 10 c.c. of the aqueous solution were treated with 5 c.c. of a reagent prepared by dissolving 0.1 grm. of 2,4-dinitrophenyl hydrazine in 1 c.c. of warm pyridine, and adding 10 c.c. of strong hydrochloric acid, the mixture being allowed to stand for fifteen minutes in a stoppered bottle, then acidified with about 7 c.c. of 2N-hydrochloric acid, shaken in a separating funnel with three portions of 19 to 20 c.c. of carbon tetrachloride, and these extracts shaken successively with 20 c.c. of 2N-hydrochloric acid. Finally, the combined carbon tetrachloride extract was diluted to 60 c.c., and its transmissive index at 435μ determined and compared with that obtained with a standard carbon tetrachloride solution of pure acetone-dinitrophenyl-hydrazone. C. A. M.

Photometric Turbidimeter. W. G. Bowers and J. Moyer. (*J. Biol. Chem.*, 1920, **42**, 191-198.)—An apparatus is described for comparing the light passing through a turbid suspension with a similar light reduced by a series of standard glasses. It consists of a closed box divided at right angles to its axis into five compartments. The two end compartments each contain an electric lamp, from which the light passes through a small hole into the next chamber. One of the chambers contains a parallel-sided glass cell 2 inches in thickness, in which the suspension is placed, the other being fitted with two revolving wooden wheels which support the standards of ground glass. The central compartment is provided with a grease spot illuminated on one side by light which has passed through the suspension, and on the other by light reduced in intensity by the standards. Two small mirrors are fitted close to the grease spot, and the images of this are viewed through a central tube fitted in the side of the box.

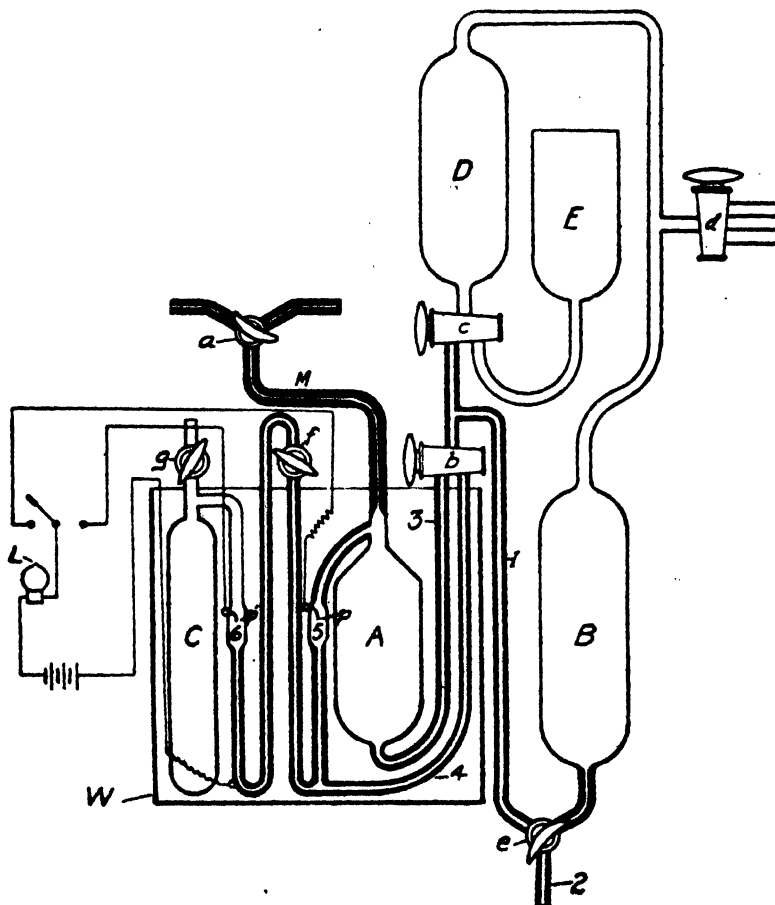
The apparatus is used by placing the suspension, prepared according to specified conditions, into the cell and revolving the standard wheels, one of which reads units and the other tenths, until both sides of the grease spot are equally illuminated.

The values of the standards in use are then read off from the upper portion of the wheels which project above the top of the box.

Tables giving series of measurements made with a barium sulphate suspension show that the greatest error between five different observers amounted to 0.075 per cent. Each substance estimated in suspension must have its own values worked out on the scales by preliminary experiments before measurements can be made. The authors find considerable practice necessary before coloured suspensions can be accurately compared with white light. T. J. W.

Weighing Burette for Gas Analysis. E. R. Weaver and P. G. Ledig. (*J. Amer. Chem. Soc.*, 1920, **42**, 1177-1185.)—The apparatus shown in the diagram

enables small amounts of gas to be accurately measured. When ready for use the measuring bulb A, and the tubes 1, 3, 4, and 5, are completely filled with mercury, and the tube 6 filled nearly to the contact point, p^1 . Mercury can be drawn into or forced out of the bulbs B or D through the stopcocks c and e by means of a small motor-driven pump. When the gas is saturated with water vapour a drop of water is introduced into the compensating vessel C, and the walls of the burette are



moistened. In making a gas measurement, mercury is run into the tube 6 from D, so as exactly to close the contact p^1 . The cocks f and g are then closed, confining a definite volume of gas in the compensator C, the bulb A is connected by means of a with the vessel containing the gas, and mercury withdrawn into D until the surface separating the gas from the confining liquid reaches M. The stopcock c is then closed, e turned to connect A and B, and the gas drawn into A, while the displaced mercury flows into B. The cock a is closed when the surface of separation between the gas and confining liquid reaches M. Mercury is now run from B into A until the pressure of gas in A is adjusted to equal that in C. Mercury is first drawn into B

through 4 until the contact point in 5 is uncovered, and then forced back again until the contact is just closed, as shown by the electric light L. The amount of mercury in the compensator between points 5 and 6 is thus adjusted. The stopcock *b* is next closed to tube 4 and opened to tube 3, and the cock *f* opened; and by running mercury between A and B through tubes 1 and 3, the pressure in A is adjusted until the contact in 5 is again just closed. Assuming the contact points *p* and *p*¹ to be on the same level, the pressure of the gas in A will be exactly equal to that in the compensator C, and its volume exactly equal to that of the mercury in B. By connecting B with tube 2 by turning *e*, the mercury is drawn off into a small flask in which it is weighed, and, from the weight, the volume of the gas in A is calculated. The parts of the apparatus which require to be maintained at constant temperature are immersed in a circular water-trough, W. C. A. M.



REVIEW.

THE IDENTIFICATION OF ORGANIC COMPOUNDS. By G. B. NEAVE and I. M. HEILBRON. Second Edition. Pp. 88. London: Constable and Co., 1920. Price 4s. 6d. net.

This small volume is intended as a practical handbook for the use of advanced students engaged in the identification of the simpler organic compounds.

The first chapter is devoted to preliminary tests, and this is followed by three pages giving the reactions for the elements. It will be noticed that no mention is made of the production of sodium thiocyanate (which may be tested for by means of ferric chloride) when a compound containing both nitrogen and sulphur is fused with sodium.

Chapter III. describes the tests employed for the detection of organic radical groups, and in this connection it may be pointed out that in the test for methoxy and ethoxy groups any compound will produce a precipitate in the alcoholic silver nitrate solution, since the hydriodic acid itself will volatilise under the conditions described.

The remainder of the book contains the reactions, melting-points, and boiling-points of a variety of substances arranged under the headings of hydrocarbons, ethers, halogen compounds, etc., and amounting to a total of between five and six hundred individual compounds. As the users of the volume are assumed to have had experience in organic preparations, very few practical details are given. A brief appendix gives a description of a convenient m.-pt. apparatus and methods for the preparation of certain special reagents.

An examination of the figures given for the melting-point and boiling-point, and also the numbers of pages furnished in the two indices, has failed to detect errors.

The authors have succeeded in producing a concise and reliable handbook which may be thoroughly recommended to students and others engaged in the identification of organic compounds.

T. J. WARD.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE ESTIMATION OF CODEINE.

By H. E. ANNETT, D.Sc.(LOND.), F.I.C., AND HARIDAS SEN, M.Sc.(CAL.)

THE absence of methods capable of accurately estimating small amounts—e.g., a few milligrams—of a particular alkaloid in the presence of large amounts of plant material is no doubt the reason why so little really satisfactory work has been carried out bearing on the manner in which alkaloids are produced in the actual plant. In our work on the Indian opium poppy we have, therefore, paid considerable attention to methods of estimation of the three chief opium alkaloids—morphine, narcotine, codeine. As a result we now claim to have devised methods which are capable of the accurate estimation of the small amounts of narcotine or codeine occurring in poppy capsules. The methods will also accurately estimate the amount of either of these two alkaloids in opium. We have not so far met with the same amount of success with morphine. In addition to our need for methods for the actual estimation of the above-named alkaloids in the actual plant, it was an urgent matter for us to have methods which were rapid, since we are engaged in collaboration with Mr. H. M. Leake, Economic Botanist to the Government of the United Provinces in plant-to-plant selection work, having for its object the production of races of plants rich in a particular alkaloid. The methods we have devised are far more rapid in their execution than any hitherto published. They further possess the advantage that they can be used when only small amounts of material are available.

The published methods for codeine estimation are those of P. van der Wielen, (*Pharm. Zeit.*, 1903, 48, 267, and *Bull. Soc. Pharmacol.*, 1910, 17, 59-63), Caspari (*Apoth. Zeit.*, 1904, 19, 874), and Andrews (*ANALYST*, 1911, 36, 489). The last-mentioned, in his paper referring to the two former methods, states: "These are both unsatisfactory, for in Van der Wielen's method the separation of codeine and narcotine depends on the complete crystallisation of the latter from an alcoholic solution of the mixed crude alkaloids, and in Caspari's method there is loss of codeine owing to its being carried down with morphine when this alkaloid is precipitated by ammonia from an aqueous liquid containing the two alkaloids." Andrews publishes

no experimental evidence to show that Van der Wielen's or Caspari's method gives unsatisfactory results. We in our turn have tested Van der Wielen's and Andrews's methods with pure alkaloids. As shown below, they gave such low results in our hands as to be useless for our purposes. A further objection to Andrews's method is the long time taken over the analysis. Caspari's process is also long, and 50 grms. of opium are required for a determination. We therefore did not examine it.

EXPERIMENTALS—Test of Andrews's Method using Pure Codeine.—The codeine used in this experiment had been twice recrystallised from alcohol and dried at 110° C. to constant weight. Three portions of 0.4, 0.2, and 0.1 gm. respectively were weighed out. Each portion was dissolved in 100 c.c. of water containing 1 c.c. of 10 per cent. of H_2SO_4 . The first part of Andrews's method consists of extracting the opium with water to dissolve out the codeine. In the case of the pure alkaloid we had, of course, to use a small amount of acid to bring the alkaloid into solution, and since opium extracts are acid it was necessary that our extract should be acid for the sake of comparison. In each case to the 100 c.c. of codeine solution 20 c.c. of a 20 per cent. solution of lead acetate were added, and from this point onward the method was the same as described by Andrews. The results obtained are summarised in the table :

Weights of Codeine taken (Grm.).	0.400	0.200	0.100
Weight of codeine theoretically recoverable* ...	0.333	0.167	0.083
Weight actually recovered, gravity figure ...	0.1616	0.1040	0.0344
" " " volume figure ...	0.1506	0.0793	0.0333
Percentage of codeine recovered on amount taken (volume figure) ...	45.2	47.6	39.9

Less than half the codeine has, therefore, been recovered in each experiment. The codeine is finally recovered by Andrews by extracting a solution made alkaline with caustic soda, four times with its own bulk of ether. The ether is then washed with water to remove caustic soda. In view of our low recovery, we again submitted the alkaline solution to four extractions with ether, with subsequent washings of the ether, and obtained 0.0412, 0.0316, and 0.0134 gm. respectively of a waxy residue. This residue was not codeine, however, since it was a neutral substance and gave no titration value when titrated with sulphuric acid. It seemed to us probable that when the ethereal solution of codeine is washed with water much loss of codeine might take place, because both codeine and ether are appreciably soluble in water. Thus Allen ("Commercial Organic Analysis," vol. vi., p. 463) states that one part of codeine is soluble in 120 parts of cold water. We have therefore tested this point and the results of our experiments show that it is obvious that the washing of the ethereal solution of codeine with water must result in appreciable loss of the alkaloid, even if only small quantities of water are used for washing purposes. We have made other determinations with Andrews's method both with pure codeine alone and in the presence of

* After addition of 20 c.c. of lead acetate solution to 100 c.c. of aqueous extract containing the codeine, Andrews filters and takes 100 c.c. of the filtrate only for subsequent recovery of codeine.

other alkaloids and also in opium. We have always obtained low results, and, moreover, our final product was not pure codeine.

Tests with Van der Wielen's Method using Pure Alkaloids.—In Van der Wielen's process (*loc. cit.*) both codeine and narcotine are estimated in the same sample. We have tested the process with pure codeine alone, pure narcotine alone, and with mixtures of pure codeine and narcotine. We followed the method as laid down in Van der Wielen's later paper. Four series of experiments were started.

Series I.—Crystalline codeine dried at air temperature (22° C.). *Series II.*—Anhydrous codeine dried at 110° C. *Series III.*—Codeine + narcotine. Codeine dried at 110° C. and narcotine dried at 100° C. *Series IV.*—Narcotine alone, dried at 100° C.

In each case the sample was boiled with 100 grms. of 70 per cent. alcohol in a tared Erlenmeyer flask under a reflux condenser for one hour. After cooling, any loss in weight was made good by addition of 70 per cent. alcohol. Van der Wielen takes 10 grms. of opium with 100 grms. of 70 per cent. alcohol. After restoring any lost weight after the hour's boiling with 70 per cent. alcohol, he filters and determines the dry extract in 5 grms. of the filtrate. He then proceeds with the method by taking such a quantity of this filtrate as is equivalent to 3 grms. opium. In the case of these pure alkaloids it was sufficient for us to take $\frac{3}{10}$ of the total weight of the alcoholic solution, since the alkaloids went into solution. The quantities taken were evaporated to 3 c.c. in a small dish. In series I. and II. we obtained a yellow liquid with fine yellow suspended particles. In series III. and IV. (containing narcotine) the solutions set to a solid mass. The contents of the dish were then washed out into a flask with 7.5 c.c. of water; 90 c.c. of ether were next added, and after shaking, 5 c.c. of 10 per cent. of NaOH solution. The flask was then allowed to stand, with frequent shakings, for three hours; 3 grms. of gum tragacanth were next added to aid separation of the ether, and exactly 75 c.c. of the ethereal solution (which equals 2.5 grms. of opium in the actual method) were decanted off and evaporated to dryness. The residue was dissolved in 4 grms. of 90 per cent. alcohol and set aside for twenty-four hours. The crystals of narcotine, if any, which separated were collected on a filter, washed with 5 c.c. alcohol, dried at 100 C., and weighed. As narcotine is not entirely soluble in alcohol, a correction of 0.016 must be applied. (Note.—We found that this correction factor under our conditions of temperature, etc., was about 0.021.) The filtrate from the narcotine was diluted with 10 c.c. water and then evaporated to 10 c.c.; series I. and II. (pure codeines) give no precipitate, but with series III. and IV. (containing narcotine) instantaneous precipitation took place on addition of water, and a copious precipitate came down on concentration to 10 c.c. Van der Wielen filters off at this stage in the case of an opium analysis and discards the precipitate as a resinous one. Unfortunately we did not examine the precipitate, but we dried and weighed it. These weights were as follows: Series III.—(a) 0.0234, (b) 0.0256, (c) 0.0270 gm. Series IV.—(a) 0.0218, (b) 0.0210, (c) 0.0072 gm.

If we included this weight with that of the narcotine obtained instead of the solubility correction (0.016 gm.) laid down, the figures for narcotine recovery would in all cases but the last be fairly near the theoretical. Since we get no such precipi-

tate in the case of the pure codeine series, the presumption is that this precipitate is narcotine. The precipitate is filtered off, and the filter well washed with water. Excess of $\frac{N}{10}$ HCl was added to this filtrate and a few drops of methyl orange. After standing for fifteen minutes the excess of $\frac{N}{10}$ HCl was titrated with $\frac{N}{10}$ NaOH. It was determined by actual titration that 1 c.c. $\frac{N}{10}$ HCl = 0.0307 grm. crystallised codeine, or 0.0299 grm. amorphous codeine.

The results obtained are set out below :

Series.	Weights of Alkaloids taken.	Weight of Narcotine found (excluding Correction 0.016).	Actually present.	Per Cent. Recovery.	Weight of Codeine found.	Actually present.	Per Cent. Recovery.
		Grm.	Grm.		Grm.	Grm.	
I. Pure crystallised codeine	(a) 0.400 grm.	0.0162	0.0000	—	0.0698	0.1000	69.8
	(b) 0.200 "	0.0096	0.0000	—	0.0339	0.5000	67.8
	(c) 0.100 "	0.0048	0.0000	—	0.0165	0.0250	66.0
II. Pure amorphous codeine dried at 110° C.	(a) 0.400 grm.	0.0180	0.0000	—	0.0646	0.1000	64.6
	(b) 0.200 "	0.0092	0.0000	—	0.0345	0.5000	69.0
	(c) 0.100 "	0.0036	0.0000	—	0.0152	0.0250	60.8
III. Pure amorphous codeine + pure narcotine		(including correction 0.016).					
	(a) 0.400 grm. codeine, 0.400 grm. narcotine	0.0904	0.1000	90.4	0.0723	0.1000	72.3
	(b) 0.200 grm. codeine, 0.200 grm. narcotine	0.0384	0.0500	76.8	0.0291	0.0500	58.2
	(c) 0.100 grm. codeine, 0.100 grm. narcotine	0.0198	0.0250	79.2	0.0191	0.0250	76.4
IV. Pure narcotine dried at 100° C.	(a) 0.400 grm. narcotine	0.0884	0.1000	88.4	0.0000	0.0000	—
	(b) 0.200 grm. narcotine	0.0412	0.0500	82.4	0.0000	0.0000	—
	(c) 0.100 grm. narcotine	0.0208	0.0250	83.2	0.0000	0.0000	—

In our hands, therefore, Van der Wielen's method has proved unsatisfactory, and has only recovered about $\frac{2}{3}$ of the codeine present. Of the codeine as much as 15 to 20 per cent. is seen to have been precipitated from 90 per cent. alcohol at the

stage where narcotine should have been obtained. As regards narcotine, the method has given results rather nearer the theoretical, but it must be remembered that a big correction factor for solubility in alcohol has been largely responsible for this.

We agree with Andrews that it is unsatisfactory to use a method for estimation of codeine and narcotine in which the separation of the two alkaloids depends on complete separation of narcotine from codeine by crystallisation.

We have found, in short, that Van der Wielen's method gives such unsatisfactory results with pure alkaloids that it can hardly be of much use in opium analysis.

The following are the details of the method we have devised. The method can be used for opium, pure alkaloids, or for plant material with appropriate modifications as to amounts of material taken.

When working with opium it is preferable to dry the substance first, otherwise allowance has to be made in the subsequent calculations for water present in it. In the analysis of opium we usually take 10 grms. of the dry substance and rub it down very thoroughly in a mortar with 4 grms. of freshly slaked lime and sufficient water to make a paste. More water is then used until 100 c.c. have been added in all. The whole is well stirred at intervals during half an hour and then filtered on a Buchner funnel. To 50 c.c. of the filtrate (=5 grms. of opium) are added 40 c.c. of 2 per cent. acetic acid and 10 c.c. of basic lead acetate solution of sp. gr. 1.25. The whole is vigorously shaken and filtered. To 75 c.c. of the filtrate (=3.75 grms. opium) are added 2 grms. of freshly slaked lime, and the flask is then shaken at intervals during half an hour and the contents filtered; 50 c.c. of the filtrate, representing 2.5 grms. of opium, are then shaken with three successive portions each of 50 c.c. of toluene. The combined toluene extract is filtered and 120 c.c. (2 grms. of opium) are placed in a distilling flask, and dry hydrochloric acid gas is cautiously bubbled through it. Codeine hydrochloride separates instantly as a white flocculent precipitate. Care must be taken to avoid great excess of hydrochloric acid; we usually pass a steady stream of gas for one minute. A stream of air is next passed through the flask for ten minutes to remove excess of acid. The toluene is then filtered into a fresh distilling flask, and hydrochloric acid gas is again passed through it in order to see if the precipitation of codeine hydrochloride is complete. If a further precipitate is obtained, the toluene is again aerated, and then filtered through the same filter-paper. After experience in the method we find that all the codeine can be separated by the first treatment with hydrogen chloride. The flasks and filter-paper are then washed with small quantities of water through the filter into a small weighed glass dish, 10 to 15 c.c. of water being usually sufficient. This is then evaporated on the water-bath, fine clusters of crystals being obtained. The dish is finally dried in the water-oven and weighed. The gravimetric figure may also be checked by a chlorine estimation, but in view of the low chlorine content of the hydrochloride we place greater reliance on the gravimetric figure.

The final product is usually somewhat coloured, but experience shows that the amount of colouring matter is of no significance, and it can be entirely separated if

desired by adding ammonia to the aqueous solution, and allowing the latter to stand over-night. On filtration the codeine can be separated by extracting with toluene. The hydrochloride is precipitated as before by gaseous hydrogen chloride.

For rapid estimations quite good results can be obtained by dispensing with the basic lead acetate treatment. The final product is then strongly coloured, but it seems that the colouring matter does not introduce an error greater than about 5 to 10 per cent. It can be removed fairly completely with animal charcoal with only slight loss of alkaloid.

The method depends on the fact that when opium is treated with slaked lime and water all the morphine and codeine pass into solution. The codeine can then be completely separated from this lime solution by agitation with toluene; the morphine, by virtue of its phenolic hydroxyl group, is held in solution in combination with the lime, whereas the codeine forms no such compound. A study of the literature and our own experience shows that no appreciable quantity of any other alkaloid passes into our final product. Narcotine we have shown (ANALYST, 1918, 43, 205) to be completely insoluble in lime and water. Narceine is stated to be soluble in alkalis (Henry, "Plant Alkaloids," p. 242), but it is insoluble in toluene. Papaverine is insoluble in alkalis (Pictet, "Vegetable Alkaloids," p. 286), and so does not influence the method. Thebaine is also insoluble in alkalis (*ibid.* p. 283). The remaining alkaloids are present in such small amounts in opium that they cannot affect the method in any case. We have shown (*loc. cit.*) that 50 c.c. of water in the presence of 1.25 grms. of slaked lime are capable of dissolving 0.38 gm. of codeine. In the B.P. (1914) method for the estimation of morphine in opium the quantities used are 8 grms. opium, 2 grms. slaked lime, and 80 c.c. water. These amounts of lime and water would dissolve about 0.61 gm. of pure codeine. In 8 grms. of opium this represents a percentage of 7.6. We have, so far, not had an opium containing as much codeine as this, but if one should be found the method could still be used by taking only half the quantity of opium laid down by us.

It will be noticed that after making the lime solution we acidify with acetic acid and add basic lead acetate. We find that this procedure removes a number of substances, notably colouring matters, which, in the case of Indian opium at least, are apt to contaminate the codeine hydrochloride precipitate. Basic lead acetate removes no codeine or morphine. The filtrate from the lead precipitate was, of course, treated with lime, in order to set the codeine free again for extraction with the toluene. The toluene extract, as far as we have been able to investigate, contains only very small amounts of substances other than codeine. We tried to estimate the codeine in the toluene extract polarimetrically, but the results were not satisfactory.

After various attempts to separate the codeine in a pure form, we found that the most satisfactory results could be obtained by isolating it as hydrochloride. Beal and Brady (ANALYST, 1916, 41, 132), working with coniine, state that they obtained an oil as well as the crystals, and assumed the oil to be a higher hydrochloride. We also, in the case of codeine, frequently obtained an oil by passing in too great an excess of hydrochloric acid gas. The codeine hydrochloride we recover is in a

crystalline form, occasionally contaminated with some slight amount of colouring matter.

The method has been tested with pure codeine in varying amounts. Samples of opium with and without known added amounts of codeine have been examined by it. We have also carried out Andrews's method and our method on the same sample of opium. Andrews's method has given in our hands only 40 to 50 per cent. of the codeine which our method gave in the same samples. Our experiments with Andrews's method in pure codeine recovered only 40 to 46 per cent. of the codeine taken. This is a further indication of the correctness of our method.

EXPERIMENTS WITH PURE CODEINE.—Six portions of 0.05, 0.10, 0.20, 0.30, 0.40, and 0.50 grm. of pure crystalline codeine were each triturated in a mortar with 2 grms. of lime and 80 c.c. of water. They were stirred at intervals during half an hour, and the contents filtered. In each case 50 c.c. ($=\frac{5}{8}$ of the total quantity taken) were placed in a 100 c.c. flask, together with 40 c.c. of 2 per cent. acetic acid, and 0.5 grm. dry basic lead acetate, and the contents made up to 100 c.c. After shaking and filtration, 80 c.c. of each filtrate was then shaken up with 2 grms. freshly slaked lime and filtered. Fifty c.c. ($=\frac{5}{8}$ of the original quantity taken) were then extracted with three successive portions of 50 c.c. of toluene, and 120 c.c. of the filtered toluene extract were then taken for separation of the codeine as hydrochloride. The following results were obtained:

Weight of Crystalline Codeine taken.	Weight of Codeine Hydrochloride corresponding with Five-Sixteenths of the Codeine taken.	
	Actual Yield.	Theoretical Yield.
0.050 grm.	0.0148 grm.	0.0143 grm.
0.100 "	0.0302 "	0.0286 "
0.200 "	0.0588 "	0.0572 "
0.300 "	0.0934 "	0.0858 "
0.400 "	0.1110 "	0.1114 "
0.500 "	0.1472 "	0.1430 "

Allen ("Commercial Organic Analysis," vol. ii., p. 391) states that the composition of codeine hydrochloride after drying at 100° C. is $C_{18}H_{21}NO_3 \cdot HCl, \frac{1}{2}H_2O$. This corresponds with our experience, and we have used this formula in our calculations.

EXPERIMENTS WITH OPIUM WITH AND WITHOUT KNOWN ADDED AMOUNTS OF CODEINE.—Four portions, each of 8 grms., of finely powdered air-dried opium were weighed, and to the second, third, and fourth portions respectively were added 0.04, 0.08, and 0.16 grm. of pure codeine. Each portion was then triturated with 4 grms. of lime, and a little water as described, and more water was added, until 80 c.c. had been added in all. After filtration, 50 c.c. were taken for addition of 40 c.c. of 2 per cent. acetic acid and 10 c.c. of basic lead acetate solution. Seventy-five c.c. of the filtrate from this treatment were shaken with lime, and 50 c.c. of the resulting filtrate were extracted with three successive portions of 50 c.c. of toluene. Of the

combined filtered toluene extract, 120 c.c. were in each case taken for separation of the codeine hydrochloride. The following results were obtained :

Opium taken.	Codeine added.	Codeine Hydrochloride.	
		Actual Yield.	Theoretical Yield* on Basis of Expt. No. 1.
8 grms.	Nil grm.	0.0984 grm.	— grm.
8 "	0.04 "	0.1148 "	0.1105 "
8 "	0.08 "	0.1275 "	0.1226 "
8 "	0.16 "	0.1407 "	0.1468 "

CONCLUSION.—The method we have devised for codeine estimation gives accurate results. It has the advantage that the codeine can be isolated in a pure crystalline form as hydrochloride. The method is, moreover, rapid and easy of execution.

We have made use of a modification of the method for the estimation of codeine in plant material such as poppy capsules with eminently satisfactory results. It is hoped later to make a further communication on this work.

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THE INFLUENCE OF THE FREE FATTY-ACID CONTENT IN THE VALUATION OF CHINESE WOOD-OIL BY THE BROWNE POLYMERISATION TEST.

By PHILIP E. JAMESON.

In testing Chinese wood-oil ("tung-oil") for varnish-making purposes, advantage is taken of the remarkable property of the oil to solidify at elevated temperatures. This phenomenon is due to polymerisation, and, as far as is known at present, is not characteristic of any other glyceride.

The Browne method (ANALYST, 1912, 37, 410) is the one commonly used by varnish chemists in this country, and is officially adopted by the American Society for Testing Materials (Standard Specification for Raw Tung-oil, D. 12-16). Briefly, it consists in heating 5 c.c. wood-oil in a test-tube immersed in an oil-bath kept at a constant temperature of 540° F. By means of a glass rod placed in the sample the operator judges when the oil has solidified completely. This should not take more than twelve minutes for a pure oil. Browne (*loc. cit.*) showed that on mixing pure wood-oil with soya-bean, cotton-seed, and other oils, so that the mixture contained 10 per cent. foreign oil, the time required for polymerisation was increased from

* 0.100 grm. codeine = 0.121 grm. $C_{18}H_{21}NO_3 \cdot HCl \frac{1}{2} H_2O$.

two and a half to three minutes. This has led to the idea that when an oil fails to pass the twelve-minute limit it is necessarily adulterated with one of these foreign oils.

In reviewing the literature of chinawood-oil testing, the writer noticed that none of the investigators has taken into consideration the influence of the free fatty-acid content on the time of polymerisation. In fact, very few chemists have even stated the percentage of free acid present in the oils under examination.

In the course of testing several hundred samples of wood-oil in this laboratory, it was found that the free acid content has a direct influence on the time required for polymerisation—in other words, since the fatty acids do not themselves possess the property of solidifying with heat, it follows that the time of polymerisation is increased in proportion to the amount of free fatty acid present in the oil.

The time of polymerisation of the majority of wood-oils available in the present market exceeds the twelve-minute limit. In order to eliminate the influence of the free acid, the time of polymerisation of oils from which the acid had been entirely removed was determined. Several methods were tried for removing the acid without otherwise changing the oil. The following was found to give the best results: 5 grms. dry calcium hydroxide of at least 75 per cent. strength are added to 100 c.c. wood-oil, stirred well at intervals for fifteen minutes, and then filtered through a 25 cm. "quantitative" filter-paper on a 12 cm. "ribbed" funnel. The oil so obtained will be clear and lighter in colour than the original, and should be entirely free from fatty acid. The time of polymerisation is now determined and compared with that of the original.

The results of the examination of a number of samples from different importers are shown in the table:

Sp. Gr. at 60° F.	Percentage Free Fatty Acid.	Time required for Polymerisation at 540° F.	
		(a) Original Oil.	(b) Acidless Oil.
0.940	1.9	13 mins.	12 mins.
0.940	2.8	13½ "	12 "
0.939	4.6	14½ "	12 "
0.939	4.5	14½ "	12 "
0.940	2.8	13 "	11½ "
0.939	3.7	13½ "	12 "
0.939	3.8	13½ "	12 "
0.940	2.4	13 "	11½ "
0.940	2.7	13 "	11½ "
0.940	3.2	13½ "	11½ "
0.940	3.3	13 "	11½ "
0.939	3.4	13½ "	12 "

SPECIFIC GRAVITY.—Determined at 60° F. by means of a Westphal balance.

PERCENTAGE FREE FATTY ACID.—Twenty-five grms. oil are weighed into a 500 c.c. flask and shaken with a mixture of 100 c.c. redistilled alcohol and 100 c.c. redistilled neutralised benzol. The mixture is then titrated with $\frac{N}{2}$ potassium hydroxide solution, with phenolphthalein as indicator, until

the red colour remains after stoppering the flask and shaking. The results are calculated to percentage of eleostearic acid, 1 c.c. $\frac{1}{2}$ KOH being equivalent to 0.056 grm. eleostearic acid, M.W. 280.

HEATING TEST.—The Browne method (*loc. cit.*) is used as originally published with the exception of the following modification: Instead of the 6 cm. by 12 cm. oil-bath a larger one, 12 cm. by 12 cm., was used. This enables the operator to maintain a more uniform temperature, and at the same time permits the use of three tubes instead of one, the first being used for the sample "as received," the second for the acidless oil, and the third for the "standard." The latter is a wood-oil of known purity which serves as a check on the results. (The standard used by the writer contained 0.5 per cent. free acid and polymerised in eleven minutes at 540° F.)

The figures in the table show the variation in amount of the free fatty acid. After testing several hundred samples of wood-oils by this method, few samples were found which failed to polymerise in twelve minutes or less following the removal of the free fatty acids.

The fact that most oils which fail to pass the twelve-minute requirement "as received" will do so when rendered neutral supports the conclusion that such oils are not necessarily adulterated but contain too much free acid for varnish-making purposes, since the free acid is more deleterious than foreign oil. This matter of excessive free acid should receive more consideration from exporters in the future. The high acid content, as well as the dark colour of most of the oils in the market to-day, is no doubt due to the practice the Chinese producers make of heating the kernels before pressing. A prime wood-oil for varnish-making purposes should not contain over 1 per cent. free acid.*

The author hopes that the use of this modification of the Browne heating test will enable chemists to judge more accurately the quality of wood-oils in the future and revise specifications accordingly. The proportion of tri-eleostearin, to which the phenomenon of polymerisation is due, undoubtedly varies in oils from different localities according to soil conditions, botanical variation, etc.

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APPARATUS FOR EVOLUTION METHODS OF ANALYSIS.

By E. R. DOVEY.

THE apparatus shown in Fig. 1 was found to be exceedingly convenient for the examination of pyrolusite, and has been since used for various evolution methods, such as the estimation of sulphur in steel by the ammoniacal cadmium sulphate process.

The diagram is self-explanatory: the bulk of the absorbing liquid is placed in the large test-tube C, a smaller quantity in the U-tube E. It is easy to charge, and

* The American Society for Testing Materials Specification for Raw Tung-oil, mentioned above, allows a maximum of 3 per cent. free fatty-acid. The writer has seen very few wood-oils of this high acidity which would pass the twelve-minute limit with the heating test.

is proof against sucking back, as on the slightest diminution of pressure in the flask A air bubbles in through the tube B. It is found in practice that in most cases complete absorption takes place in the tube C, which at the end of the distillation is very easily detached and washed out.

The apparatus shown in Fig. 2 was primarily designed for the estimation of carbon in steel by the wet combustion method, absorbing the CO_2 in barium hydroxide solution, and afterwards titrating the barium carbonate. It, however, has been used for other evolution methods where it is desirable to have a condenser above the boiling flask. It consists of a boiling flask, A, fitted with a short Liebig

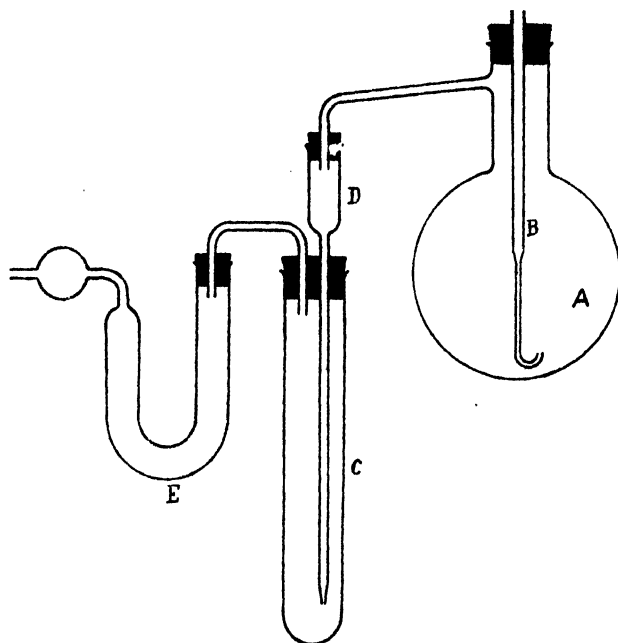


FIG. 1.

condenser, B, through which passes the long stem of a tap funnel, C. The whole is connected, as shown, with a 100 c.c. burette containing a Young's rod and disc, which fits loosely, and extends about two-thirds of the length of the burette. The top of the latter is attached by a three-way tap to an aspirator. For the carbon estimations, after the apparatus has been washed out with a stream of CO_2 -free air, 30 c.c. of barium hydroxide solution are sucked up into the burette, the flask A charged, and the estimation proceeded with, controlling the flow of gas by means of the burette stopcock, sufficient head of water being maintained in the aspirator all the time to prevent any escape of the barium hydroxide through the lower tap. When the absorption is complete, the tip of the burette is passed through a rubber stopper fitted into a wide filter-tube, the liquid run straight from the burette through the filter, the burette and precipitate being washed by allowing the filter-pump to draw

portions of the aspirator water through the apparatus. In this way the barium carbonate can be filtered off and washed without any contact with the atmosphere.

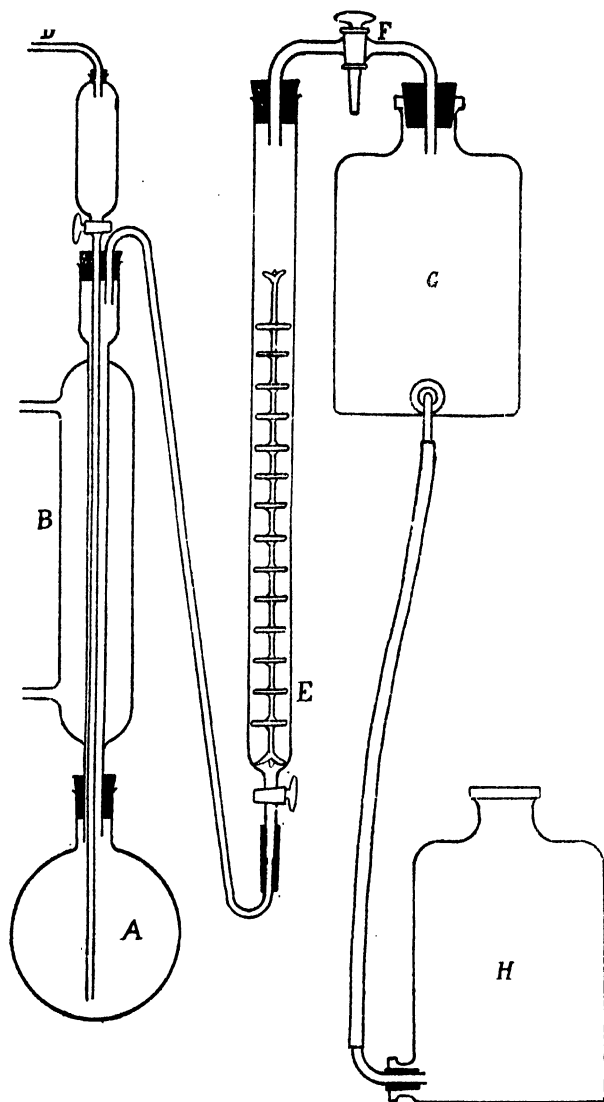


FIG. 2.

The burette fitted with the rod and discs forms a very efficient absorption apparatus, and may be used for many purposes.

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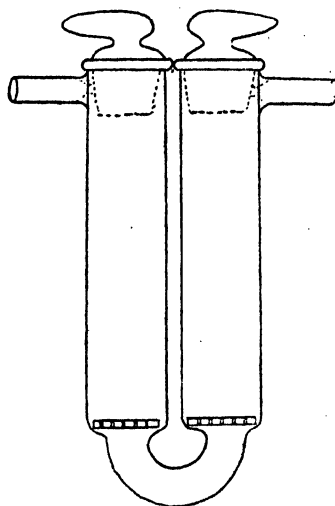


AN IMPROVED FORM OF U-TUBE.

By E. R. DOVEY.

THE form of U-tube shown in the sketch has been found to be more easily cleaned and charged, and also much less fragile than the ordinary form. The two limbs are brought together so that the rims at the top are in contact. A slight squeeze when holding the tube during charging or connecting is much less likely to result in fracture than in the ordinary form. Two perforated Gooch discs rest at the bottom of each limb, keeping the bent portion free from absorbent material.

GOVERNMENT LABORATORY,
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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Estimation of Caffeine in Coffee Mixtures and so-called Caffeineless Coffees. E. Vautier. (*Ann. Chim. anal. Appl.*, 1920, 2, 168-172.)—The method of estimating caffeine previously described (*ANALYST*, 1918, 43, 410) gives sufficiently accurate results in the analysis of ordinary coffees, but in the case of coffees or mixtures poor in caffeine it is necessary to eliminate the sources of error in the sublimation process, either by estimating the nitrogen in the crude product or by purifying the residue of alkaloid. In the first method the crude caffeine is heated in a Kjeldahl flask with 10 c.c. of sulphuric acid, 5 grms. of potassium sulphate, and 0.5 gm. of crystallised copper sulphate, and the ammonia distilled, Congo red being used as indicator. A blank estimation should be made under the same conditions. Each 1 c.c. of $\frac{N}{10}$ acid corresponds with 0.00485 gm. of anhydrous or 0.00530 gm. of hydrated caffeine. In the second method the solution of crude caffeine is evaporated to dryness on the water-bath with 0.1 to 0.2 gm. of sodium carbonate, and the residue repeatedly treated with small portions of chloroform, which does not dissolve the sodium salts of the humic acid-like impurities. The united filtrates from the insoluble residue are evaporated to dryness and the purified caffeine dried at 100° C. A caffeineless coffee yielded 0.13 per cent. of caffeine by the sublimation method, 0.05 per cent. calculated from the nitrogen, and 0.05 to 0.06 per cent. after purification with chloroform.

C. A. M.

Oxalic Acid in Rhubarb and the Removal of Oxalic Acid from Rhubarb Products. J. Angerhausen. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1920, **39**, 81-87.)—Rhubarb stalks contain 0.25 per cent. and rhubarb leaves 0.50 per cent. of oxalic acid. The latter is not present in the free state, but as alkali and calcium salts, the alkali salts predominating. The whole of the latter pass into solution when rhubarb stalks are boiled with water. When the leaves are cooked for consumption, the water in which they are boiled should be rejected, and it is advisable to add calcium carbonate (about 1 per cent. of the weight of the leaves) during the cooking operation. W. P. S.

Pentosans as a Basis in Determining the Milling Grade of Flour. J. Gerum. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1920, **39**, 65-69.)—Estimation of pentosans in flour is of little use in determining its grade; "82 per cent." flour yields 7.47 per cent. of phloroglucide and "94 per cent." flour 8.77 per cent. The starch and ash contents of the flour afford more trustworthy evidence for the purpose. W. P. S.

Identification of Sulphonal and Trional. W. Zimmermann. (*Apoth. Zeit.*, 1920, **35**, 27; through *Chem. Zeit. Rep.*, 1920, **44**, 176.)—An odour of mercaptan is observed when 0.1 gm. of sulphonal or trional is fused with 0.1 gm. of sodium salicylate and the mass then boiled with water; if 5 drops of alcohol and 5 drops of concentrated sulphuric acid are added, followed by a further 5 drops of the acid after one minute, and the mixture then warmed, a turbid red-coloured solution is obtained having an odour of methyl salicylate. A violet-coloured residue is produced when 0.2 gm. of either substance is ignited in a porcelain basin; the residue dissolves in a drop of water giving a violet-coloured solution, the colour changing rapidly to brown. The addition of a drop of hydrochloric acid produces a yellow colour, the separation of a brown precipitate and liberation of sulphur dioxide. Santonin yields a red coloration when heated with sodium salicylate. W. P. S.

Paraguay Tea. C. R. Hennings. (*Ber. Deut. Pharm. Ges.*, 1920, **30**, 22-26; through *Chem. Zeit. Rep.*, 1920, **44**, 179.)—Analysis of Paraguay tea (maté) yielded the following results: Water, 9.00; water extract, 33.10; ash in water extract, 3.8; alkaloids, 2.1; tannin, 9.79; total ash, 6.62; soluble ash, 2.26; silica, etc., 1.44; alkalinity of ash (as K_2O), 0.69; crude fibre, 15.45; ether extract, 9.8; volatile extract, 2.05; total nitrogen, 2.17; resins, 9.1 per cent. W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Reaction of Benzoic Acid and its Application to the Toxicological Detection of Atropine, Cocaine, and Stovaine. M. Guerbet. (*Comptes rend.*, 1920, **170**, 40-41.)—On treating benzoic acid with fuming nitric acid (sp. gr. 1.49), it gives a mixture of *o*-, *m*-, and *p*-nitrobenzoic acids, and these when heated with stannous chloride are reduced to *o*-, *m*-, and *p*-aminobenzoic acids, which when treated with nitrous acid in hydrochloric acid solution are converted into the corresponding

diazotic chlorides. Lastly, these chlorides combine in ammoniacal solution with β -naphthol to form the isomeric *o*-, *m*-, and *p*- β -naphtholazobenzoic acids, in the form of an orange-red precipitate. The reaction is also given by benzoates, and compounds, such as cocaine and stovaine, which contain the benzoyl radicle, or which, like atropine, yield benzoic acid on oxidation. In applying the test a minute quantity of the substance (*e.g.*, the extract obtained in Stas' method) is treated with 3 or 4 drops of fuming nitric acid on a clock-glass and the liquid evaporated to dryness on the water-bath. The residue is treated with 1 drop of a 10 per cent. solution of stannous chloride, heated for two or three minutes, cooled and treated with 2 drops of sodium nitrite solution (1 : 100). Finally 3 or 4 drops of a 1 per cent. solution of β -naphthol in 10 per cent. ammonia solution are added, and the formation of a reddish-orange precipitate will indicate the presence of benzoic acid. The reaction is obtained very distinctly with 0.1 mgrm. of the above-mentioned alkaloids.

C. A. M.

Biochemical Study of Copra Meal. F. O. Santos y Alvarez. (*Philippine J. Sci.*, 1920, 16, 181-189).—Investigation of the nitrogen distribution in that portion of copra meal insoluble in hot water showed that the meal is rich in the amino-acids necessary for the maintenance of growth; arginine, histidine, and cystine are present in considerable quantity, but the amount of lysine is small. Only 47.55 per cent. of the total protein nitrogen is soluble in 0.2 per cent. potassium hydroxide solution; in the case of lupine seeds, 96 per cent. of the protein nitrogen is soluble in alkali solution. Feeding experiments with copra meal indicated that when fed alone the meal cannot promote growth in pigs, but that when mixed with green leaves it furnished a fairly perfect fodder.

W. P. S.

Volumetric Method for the Determination of the Diastatic Capacity. J. T. Flohill. (*J. Ind. and Eng. Chem.*, 1920, 12, 677).—One c.c. of a 5 per cent. malt extract prepared by Lintner's method is introduced into 100 c.c. of a 2 per cent. solution of soluble starch in a 200 c.c. flask, and the mixture allowed to stand for one hour at 20° C. The liquid is then treated with 10 c.c. of $\frac{N}{10}$ sodium hydroxide solution and made up to 200 c.c. Twenty-five c.c. are transferred to an Erlenmeyer flask, treated with 10 c.c. of each of the Fehling solutions, and the volume made up to 50 c.c. The mixture is boiled slowly for two minutes, and immediately cooled in running water, but not much below 25° C. Ten c.c. of 30 per cent. potassium iodide solution, or 3 grms. of potassium iodide and 10 c.c. of 25 per cent. sulphuric acid, are added, and the liberated iodine titrated with $\frac{N}{10}$ thiosulphate solution. A blank test is made simultaneously, and the difference between the amounts of thio-sulphate used in the two titrations is equivalent to the copper sulphate reduced by the maltose. If 25 c.c. of the above-mentioned solution reduce Fehling's solution

equivalent to m c.c. of thiosulphate solution, its strength is $\frac{m}{62.5n} \times 100 = 320 \frac{m}{n}$

degrees Lintner, where n represents the thiosulphate equivalent of the amount of iodide liberated by the copper sulphate in a blank test.

C. A. M.

Serum obtained by the Spontaneous Curdling of Milk. Its use in detecting Adulteration. J. Grossfeld. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1920, 39, 140-145.)—The serum obtained by allowing milk to curdle spontaneously may be used for the usual investigations applied to sera; it possesses the advantage that no foreign substance is introduced, and the only correction necessary is that for the effect of the acidity on the specific gravity. For each c.c. $\frac{N}{10}$ alkali solution used in neutralising 10 c.c. of the milk, 0.00011 is added to the specific gravity found.

W. P. S.

Estimation of Tyrosin and Dibasic Amino Acids in Yeast Proteins. P. Thomas and A. Chabas. (*Comptes rend.*, 1920, 170, 1622-1625.)—The colorimetric methods of estimating tyrosin are not absolutely specific for that substance, but also indicate phenolic constituents of proteins free from tyrosin. By the method of Folin and Denis (*J. Biol. Chem.*, 1912, 12, 245) the yeast protein, cerevisine, was found to contain 6.77 per cent., and the protein, zymocaseine, 7.54 per cent. of tyrosin. The results obtained with Millon's reagent, after eliminating sulphates, were 7.38 and 4.23 per cent. respectively. Direct gravimetric estimation of the tyrosin extracted from the products of the hydrolysis gave 4.13 per cent. for the cerevisine and 2.85 per cent. for the zymocaseine. The amounts of glutamic acid, isolated as hydrochloride and purified by fractional crystallisation from alcohol, were 6.26 per cent. for cerevisine and 0.94 per cent. for zymocaseine, whilst in each case the amount of aspartic acid was less than 1 per cent.

C. A. M.

ORGANIC ANALYSIS

Estimation of Acetaldehyde in Paraldehyde. W. Stuve. (*Apoth. Zeit.*, 1920, 35, 145; through *Chem. Zeit. Rep.*, 1920, 44, 178.)—Ten c.c. of $\frac{N}{10}$ mercuric chloride solution, 2 grms. of potassium iodide, 20 grms. of 15 per cent. sodium hydroxide solution, 50 grms. of water, and 5 c.c. of the paraldehyde are mixed, shaken occasionally during fifteen minutes, then diluted to 100 c.c. and filtered. Fifty c.c. of the filtrate are treated with 0.5 gm. of gum arabic, 5 c.c. of sodium hydroxide solution, and 3 c.c. of formaldehyde; after fifteen minutes, the mixture is acidified with 15 c.c. of dilute acetic acid, cooled, the reduced mercury is dissolved by the addition of 10 c.c. of $\frac{N}{10}$ iodine solution, and the excess of iodine is titrated with $\frac{N}{10}$ thiosulphate solution. The number of c.c. of iodine solution used by the mercury is multiplied by 2 and subtracted from the number of c.c. equivalent to 10 c.c. of the mercuric chloride solution; each c.c. of $\frac{N}{10}$ iodine solution is equivalent to 0.0022 gm. of acetaldehyde.

W. P. S.

Adsorption by Charcoal. Relation of Service Time to Adsorption and Absorption. H. H. Lowry and G. A. Hulett. (*J. Amer. Chem. Soc.*, 1920, 42, 1393-1408.)—Measurements of the adsorption of nitrogen and carbon dioxide by charcoals showed that the adsorptive capacity per gm. of charcoal at 25° C. and 760 mm. may vary as much as 100 per cent., and there appears to be no relation between service time and adsorption of a gas by various charcoals. (Service time

denotes the period elapsing before a toxic gas can be detected in the effluent air when air containing a known quantity of the gas is passed at a definite rate through a sample of charcoal of standard dimensions.) Adsorption of nitrogen and carbon dioxide by charcoal may be considered to be in a condensed layer 1 molecule deep. Measurements of the isothermal adsorption of water-vapour by charcoal at 25° C. showed that water is not adsorbed by charcoal, but is held by capillary action—i.e., is absorbed. Using Anderson's formula connecting radius of a capillary with vapour pressure, the mean diameters of the capillaries of the four charcoals used in the experiments were calculated, and found to vary between 2.8×10^{-7} cm. and 1.2×10^{-7} cm. By means of this same formula and the isothermals for water the surfaces of the charcoal were found to vary from 160 square metres to 436 square metres per grm. of charcoal. This variation in surface was not always accompanied by a corresponding variation in adsorptive capacity. No relation was found to exist between the service time of the charcoals and the volume of the capillaries; the same was true for the saturation points and retentivities, indicating that factors other than adsorption and capillary action influence the minute service of a charcoal.

W. P. S.

Estimation of Cyanamide and Dicyano-diamide in Calcium Cyanamide. Marquayrol, P. Loriette, and L. Desvergues. (*Ann. Chim. anal. Appl.*, 1920, 2, 164-167.)—The total nitrogen is estimated by Kjeldahl's method. The estimation of the cyanamide is based upon the fact that an aqueous solution of calcium cyanamide treated with nitrate or acetate of silver in presence of ammonia yields a precipitate of silver cyanamide containing the whole of the cyanamide nitrogen. The amount of cyanide is then calculated from the proportion of nitrogen estimated by Kjeldahl's method. Estimation of the silver in the precipitate, as proposed by Kappen, gives inaccurate results, owing to the composition of the precipitate varying considerably. The error is reduced by using very dilute solutions of cyanamide, silver nitrate and ammonium thiocyanate, dissolving the calcium cyanamide in the minimum amount of nitric acid, and rendering the solution slightly ammoniacal before precipitating the silver cyanamide. The estimation of the dicyano-diamide depends upon the fact that its aqueous solution treated with nitrate or acetate of silver, in the presence of potassium or sodium hydroxide, yields a precipitate containing the whole of the dicyano-diamide nitrogen. After precipitation of the cyanamide, the liquid is filtered, the filtrate boiled with a little 10 per cent. potassium hydroxide solution until all ammonia is expelled, the resulting precipitate separated and washed, and its nitrogen estimated by Kjeldahl's method. It has been shown by Caro (*ANALYST*, 1911, 36, 76) that the composition of the precipitate varies with the quantities of dicyano-diamide and silver salt used, and for this reason volumetric methods based on the estimation of the silver are inaccurate. There are also present in calcium cyanamide other nitrogenous compounds—viz., urea, up to about 0.5 per cent. of nitrogen calculated on the cyanamide; compounds insoluble in water and in nitric acid (0.5 to 1.5 per cent. of N on the calcium cyanamide); and a small quantity of soluble nitrogen not precipitated by silver.

C. A. M.

Estimation of Glycol. B. Müller. (*Chem. Zeit.*, 1920, **44**, 513-515.)—For the estimation of ethylene glycol (made and sold in Germany as a substitute for glycerol) the dichromate and acetin methods used in glycerol analysis may be used, but the acetin method is unsuitable in the case of dilute aqueous solutions of the glycol. In the dichromate method, heating for four hours is necessary for complete oxidation. The glycol may also be estimated by oxidation with chromic acid and sulphuric acid and weighing the amount of carbon dioxide produced. Since the glycol is not oxidised completely to oxalic acid by alkaline permanganate solution, Benedikt and Zeigmondy's method for glycerol cannot be applied to glycol.

W. P. S.

Comparison of Different Methods for Estimating the Iodine Value of Fats. T. Sundberg and M. Lundborg. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1920, **39**, 87-95.)—The Hanús method yields results which agree most closely with those found by the Hubl method; Wijs's method gives higher results, whilst those obtained by Winkler's method are lower than the Hubl values. In the case of linolic acid, the Hubl value lies nearer to the theoretical iodine value than does the Wijs' value. The Hanús method is preferable to the Winkler method on account of the shorter reaction period required.

W. P. S.

Detection of Oxalic Acid and Lactic Acid, especially their Differentiation from Tartaric Acid. K. Brauer. (*Chem. Zeit.*, 1920, **44**, 494.)—When heated with concentrated sulphuric acid and resorcinol, oxalic acid yields a brilliant violet coloration, whilst lactic acid and tartaric acid both give a red coloration. If dilute sulphuric acid (1 : 1, or even lower concentration) is used in the test the coloration obtained with the different acids is : Tartaric acid, yellow ; oxalic acid, green ; lactic acid, red. Solutions of the acids should be evaporated to dryness and the tests applied to the residues.

W. P. S.

Rectified Petroleum Spirit from the Toluene-Petrol Fraction of Borneo Petroleum. W. A. Silvester. (*J. Soc. Chem. Ind.*, 1920, **39**, 187-188r.)—The experiments cited do not support the conclusion of Evans (*J. Soc. Chem. Ind.*, 1919, **38**, 401r) that a further fractionation of the two main fractions of this petrol is impossible. It is shown that repeated cumulative fractionation is capable of separating the paraffinic and naphthenic constituents of the two principal fractions, notwithstanding the small differences in the boiling-points. Methylcyclohexane (hexahydrotoluene) was, in fact, separated from the fraction of b.pt. 102°-103° C., and the want of homogeneity, even in the best fraction (103°-104° C.), was obvious. The distillation apparatus used in the laboratory, with the exception of the Hempel and Lindemann columns and their modifications (*cf.* Washburn, *ANALYST*, 1920, 112), rely mainly on fractional condensation to effect the separation, and are therefore much less efficient than the column still-heads, in which there is also a pronounced interchange of heat between the rising vapour and the returning condensate, causing the "heavy" constituents of the vapour to condense out, whilst the "lighter" portions of the condensate are re-volatilised.

C. A. M.

Direct Estimation of Rubber Hydrocarbon in Raw and Vulcanised Rubber. W. K. Lewis and W. H. McAdams. (*J. Ind. and Eng. Chem.*, 1920, 12, 673-676.)—McIlhiney's method of determining the bromine value of unsaturated oils (*ANALYST*, 1895, 19, 14) has been adapted to the estimation of rubber hydrocarbon ($C_{10}H_{16}$)_n which forms an additional compound ($C_{10}H_{16}Br_4$)_n. The amount of substitution which takes place under specified conditions is determined, and twice the amount substituted is deducted from the total bromine absorbed, to obtain a measure of the true bromine addition. In the case of raw rubber, the sample is extracted with acetone to remove the resins, dried and dissolved in pure carbon tetrachloride, and the solution filtered from proteins and other insoluble substances, and evaporated, and the residue weighed. A known volume of carbon tetrachloride solution, containing about 0.2 gm. of the rubber hydrocarbon, is treated with a measured volume of a solution of bromine in pure carbon tetrachloride, so that an excess of about 150 per cent. of bromine is present, and the mixture allowed to stand for three hours in a dark room in a stoppered bottle. Ten c.c. of 3 per cent. potassium iodide solution (10 c.c. for vulcanised rubber) are then added, and the liberated iodine titrated. To determine the substitution 10 c.c. of 5 per cent. potassium iodate solution are next added, and the iodine corresponding to the hydrobromic acid titrated. A blank determination is made under the same conditions to eliminate errors due to impurities in the reagents. In applying the method to vulcanised rubber, the finely divided sample is extracted with acetone to remove resins, free sulphur, etc. The residue is dissolved by heating it for several hours with purified tetrachloroethane beneath a reflux condenser, the solution diluted with carbon tetrachloride to a definite volume, and an aliquot portion taken for the bromination. Substitution of bromine may be reduced to a minimum by titrating the brominated liquid in dim light. An estimation of combined sulphur in the rubber is made by the method of Davies (*CHEMIST-ANALYST*, 1915, 15, 4), the residue from an aliquot portion of the tetrachloroethane solution being treated with 10 c.c. of saturated arsenic acid solution, 10 c.c. of fuming nitric acid, and 3 c.c. of bromine water, the mixture evaporated to a syrup, and the sulphuric acid estimated gravimetrically. The percentage of sulphur multiplied by 2.13 gives the amount of rubber hydrocarbon combined with sulphur, whilst the amount of uncombined hydrocarbon is calculated from the bromine result.

C. A. M.

Volumetric Method for the Estimation of Reducing Sugars. A. Jonescu and V. Vargolici. (*Bul. Soc. Chim. România*, 1920, 11, 38-45.)—The method depends on the reduction of potassium ferricyanide; 1 molecule of dextrose reduces 5 molecules of ferricyanide in alkaline solution and under the conditions given. The reagent solution contains 46 grms. of potassium ferricyanide and 56 grms. of potassium hydroxide per litre and is standardised against pure dextrose. Ten c.c. of the reagent and 20 c.c. of water are boiled and titrated with a 0.5 per cent. dextrose solution, until the yellow ferricyanide is reduced completely to colourless ferrocyanide. Exactly 10 c.c. of the dextrose solution should be required. With coloured sugar solutions a few drops of picric acid may be added as an indicator; as soon as all the ferricyanide has been reduced, further addition of dextrose or other reducing sugar

reduces the picric acid and produces a red coloration. The method may be used for the estimation of sugar in urine (after clarification with lead acetate and sodium carbonate) and of lactose in milk. In the latter case, the lactose solution is prepared by treating 10 c.c. of the milk with 10 c.c. of a mixture containing 20 grms. of acetic acid and 10 grms. of picric acid per litre and filtering the mixture; 10 c.c. of the filtrate are neutralised with sodium hydroxide solution, diluted to 40 c.c., and this solution is used for the titration.

W. P. S.

Titration of Sugars. N. Schoorl. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1920, 39, 180-182.)—In using a method described previously by the author (*Zeitsch. angew. Chem.*, 1898, 11, 633) for the volumetric estimation of sugars based on the iodimetric titration of the excess of cupric salt, the proportions of Fehling solution, sugar solution, etc., prescribed must be used, otherwise the tables for calculating the amount of sugar present are useless. A reprint of these tables is given.

W. P. S.

Estimation of Thiocyanates in Ammoniacal Liquors. J. A. Shaw. (*J. Ind. and Eng. Chem.*, 1920, 12, 676-677.)—The ammoniacal liquor (500 c.c.) is treated with an aqueous solution of 3 to 5 grms. of ferrous sulphate and about 100 c.c. of 10 per cent. sodium hydroxide solution, allowed to stand overnight, and filtered, with the aid of suction, through an asbestos filter. This removes organic and inorganic substances insoluble in alkali solution and sulphides, and converts cyanides into ferrocyanides. The precipitate is washed with water containing about 30 grms. of sodium hydroxide per litre, and the filtrate and washings are heated to 60° C., slightly acidified with dilute sulphuric acid, and the ferrocyanides precipitated with 10 per cent. ferric chloride solution. The sludge is filtered off on an asbestos filter, washed with 5 per cent. sulphuric acid containing 5 per cent. of sodium sulphate, and used for the estimation of the cyanogen. The filtrate is made up to definite volume, and aliquot portions of $\frac{1}{10}$ taken. These are heated to 70° C., and treated with about 15 c.c. of 1 per cent. sulphuric acid (1 : 4), followed by a decided excess of sodium hydrogen sulphite solution, and the thiocyanate precipitated with hot 10 per cent. copper sulphate solution. The copper thiocyanate is separated after ten minutes in a Gooch crucible containing paper pulp, and washed with water at about the same temperature as the original liquid before filtration. The residue and paper pulp is then treated with 25 c.c. of $\frac{N}{1}$ -sodium hydroxide solution, the copper oxidised with bromine water, the liquid filtered, the filtrate treated with more bromine water and digested at 100° C. for an hour, after which it is acidified with hydrochloric acid, boiled to remove bromine and evaporated to dryness. The residue is treated with hot water containing a little hydrochloric acid, and the solution filtered from silica. Finally, the sulphate is precipitated with barium chloride, and the sulphur in the barium sulphate calculated into thiocyanate. The method, which is applicable to the waste liquors from ammonia stills in the coking industry, gives the true amount of thiocyanate within 0.01 gm. per litre.

C. A. M.

INORGANIC ANALYSIS.

Detection of Natural Barytes in Lithopone, etc. S. Stewart. (*J. Soc. Chem. Ind.*, 1920, 39, 188r.)—Inferior qualities of lithopone and other pigments composed essentially of zinc sulphide and barium sulphate sometimes contain natural barytes. This reduces the opacity and covering power of the pigment, and causes it to give a yellowish white when used in linoleum. The natural mineral may be detected by mixing a trace of the sample with a drop of water, drying the mixture, and examining it under the microscope with a $\frac{1}{4}$ to $\frac{1}{8}$ inch objective, the diaphragm being closed so as to form a dark background. Precipitated barium sulphate will then appear as a fine powder composed of minute crystals of uniform size, whilst in the case of natural barytes transparent irregular fragments of larger size will be seen. The test is rendered more certain by first removing the zinc sulphide by treatment with dilute hydrochloric acid and potassium chlorate, and examining the insoluble residue. C. A. M.

Titration of Chlorides with Silver Nitrate. H. W. Bolam. (*Chem. News*, 1920, 20, 292.)—In the case of a chromate, such as barium chromate, less soluble than silver chromate, the titration of the chloride with silver nitrate becomes difficult as regards the end-point of the reaction. This may be overcome by adding potassium chromate in quantity slightly larger than is sufficient to precipitate the barium radicle; the titration can then be carried out with silver nitrate as usual. The formation of a faint brown tinge, which is not discharged on shaking the flask, shows that the reaction is complete. H. F. E. H.

Electrometric Titrations with a Hydrogen Electrode. W. D. Treadwell and L. Weiss. (*Helv. Chim. Acta*, 1920, 3, 433-446.)—An apparatus is described with a hydrogen electrode, the change in the potential of which in acidimetric titrations can be measured with sufficient accuracy with a millivoltmeter. The electrode consists of a tube of unglazed porcelain, 6 mm. in diameter, the exterior of which is electrolytically coated with a thin film of gold, and then at its lower end for a length of about 7 cm. with a thin film of palladium black. For acidimetric titrations two such electrodes of equal size are combined to form an element. One is introduced into a suitable liquid ($\frac{N}{10}$ sodium bicarbonate solution, or a citrate solution with hydrogen exponent $p=5$) as a comparison electrode, whilst the other is immersed to the depth of about 2 cm. in the liquid to be titrated. The upper ends of the two electrodes are passed through a small glass bell-jar, which will form a water seal over the vessel containing the liquid. Ordinary hydrogen moistened with water is passed through the comparison electrode at the rate of 2 to 3 bubbles per second, thence through the titration electrode, and finally into the air through a capillary tube bent upwards. A short capillary tube filled with potassium sulphate connects the two electrodes. The liquid is titrated through a small opening in the bell-jar, of such size that it is completely filled by the capillary tube of the burette tap. The titration may also be effected in an open vessel, only the electrode (contained in a tube through which hydrogen is passed) being allowed to dip into the

liquid, or a tube coated inside with palladium may be used; but the results thus obtained are less accurate. In the case of weak acids the equilibrium constant may be readily calculated from the fall in potential in the course of the titration. The method is also applicable to the estimation of strong acids in the presence of weak acids.

C. A. M.

Estimation of Zirconium and Titanium in Zirconium Ores. G. E. F. Lundell and H. B. Knowles. (*J. Amer. Chem. Soc.*, 1920, **42**, 1439-1448).—The zirconium and titanium are precipitated by means of "cupferron," and the procedure recommended provides for the presence of all the elements which have been found in zirconium ores. The ore is fused with borax, the melt dissolved in hydrochloric acid, and the solution evaporated with the addition of sulphuric acid until fumes of the latter acid are evolved; after cooling the solution is diluted and filtered to separate silica and a small quantity of zirconium phosphate. This insoluble matter is ignited, treated with sulphuric and hydrofluoric acids to remove silicon, fused with sodium carbonate, the melt extracted with water to remove phosphate, and the insoluble residue fused with potassium sulphate, dissolved in sulphuric acid, and the solution added to the main solution. Interfering quantities of such elements as calcium, magnesium, potassium, sodium, lithium, copper, cobalt, manganese, zinc, and boron are removed by an ammonia precipitation followed by filtration. The ammonia precipitate is dissolved in dilute sulphuric acid, the solution treated with hydrogen sulphide to remove tin, lead, bismuth, and any remaining copper; after filtration, the filtrate is treated with tartaric acid, ammonia, and hydrogen sulphide, and filtered. The filtrate is acidified with sulphuric acid, boiled to expel hydrogen sulphide, cooled to 0° C., and treated with an aqueous solution of "cupferron," whereby zirconium and titanium are precipitated together with varying amounts of thorium, cerium, and other rare earths. The ignited and weighed "cupferron" precipitate is fused with potassium pyrosulphate, dissolved in sulphuric acid, and the solution divided into two parts for the estimation of titanium and the rare earths. Small amounts of titanium may be estimated colorimetrically, whilst large amounts are estimated by titration with permanganate solution after reduction to TiO. The rare earth oxides are estimated by a preliminary hydrofluoric acid treatment followed by an oxalate separation.

W. P. S.

APPARATUS, ETC.

Calorimetric Method for Standardising Thermometers by Electrical Energy. T. W. Richards and S. Tamaru. (*J. Amer. Chem. Soc.*, 1920, **42**, 1374-1377).—The method consists simply in supplying heat in successive small equal quantities to a calorimeter and noting the readings of the thermometers produced by each step. From the known heat capacity of the system the rise of temperature which ought to occur is readily calculated. The heat is derived from electrical energy supplied at known voltage through a definite constant resistance, and the procedure is essentially like that suitable for the determination of heat capacity where temperature changes are known.

W. P. S.

Fluoremetry. Quantitative Analysis by Comparative Fluorescence.

L. J. Desha. (*J. Amer. Chem. Soc.*, 1920, **42**, 1350-1363.)—A method of micro-analysis is described, of the same order of sensitiveness as colorimetry and nephelometry, which should prove to be generally applicable to the estimation of very small quantities of substances which are either fluorescent themselves or may be rendered so by the addition of a suitable reagent. Ultra-violet rays from a quartz-enclosed mercury arc, filtered from most of the visible radiation, are used to excite fluorescence in solutions of the substances contained in the comparison cylinders of the Kober nephelometer. The intensity of the fluorescent light thus produced, as observed in the eyepiece of the instrument, is equalised in the usual way by altering the heights of the exposed columns. Experiments with solutions of quinine sulphate in $\frac{N}{1}$ sulphuric acid (0.5 to 2 mgrms. per litre) and aniline iodeosine in $\frac{N}{10}$ alkali solution (2 to 4 mgrms. per litre) showed that the curves obtained by plotting the scale readings against concentrations are quite regular in the case of dilute solutions.

W. P. S.

Glass to Metal Joints. E. C. McKelvy and C. S. Taylor. (*J. Amer. Chem.*

Soc., 1920, **42**, 1364-1374.)—Methods are given for the construction of two types of glass to metal joints. To solder a glass tube into a steel, iron, copper, brass, or nickel tube, the inside of the metal tube is tinned and the end of the glass tube is platinised. For the latter purpose, the end of the tube is roughened with sand or emery, painted with a colloidal solution of platinum in lavender oil mixed with Burgundy pitch, and then heated to redness to burn off the organic matter and fuse the platinum into the glass. This end of the glass tube is inserted in the tinned end of the metal tube, a small quantity of zinc chloride solution is added as a flux, and the joint heated until the tin melts; it is then allowed to cool slowly. Soft glass tubing may be fused to iron or steel tubing by using a flux prepared by melting together equal weights of zinc oxide, borax, and powdered soda glass. The joints withstand sudden changes in temperature and are vacuum-tight.

W. P. S.



ANALYTICAL STANDARDS.

THE Council of the Society of Public Analysts have transmitted to the Council of the Institute a Report, abstracted below, of a Committee, consisting of Messrs. L. Archbutt, W. J. A. Butterfield, G. Nevill Huntly, and G. Rudd Thompson, appointed to consider the desirability of making provision for supplies of standard chemical substances.

The Committee is satisfied that there is a demand for analytical control standards, which is now being chiefly met by private enterprise and by the importation of the U.S.A. Bureau of Standards Samples, and that the demand appears reasonable for checking and educational rather than commercial purposes. The Committee think that every effort should be made to secure control of the supply of such standards by a responsible body representative primarily of analytical chemists, which might with advantage seek the co-operation of the manufacturing interests, the Engineering Standards Association, the U.S.A. Bureau of Standards, as well as perhaps the existing private organisation.

The Committee recommend that the Society of Public Analysts should take the initiative in forming a representative Analytical Standards Committee, the objects of which shall be :

1. To decide for which chemicals, commercial metals and alloys, and perhaps ores and certain other products, it is desirable to supply standard samples.
2. To settle how samples shall be provided and issued.
3. To take steps for securing comparative analyses of the proposed standard samples by groups of chemists engaged in the analysis of the particular class of materials to which the sample belongs.

The Analytical Standards Committee should consist of five to seven members of the Society of Public Analysts, actively engaged in analyses of pure chemicals, alloys, metals, etc.; and as the Society numbers among its members representatives of other bodies, such as the Institute of Chemistry, the Association of British Chemical Manufacturers, the Society of Chemical Industry, the Iron and Steel Institute, the Institute of Metals, etc., the Committee suggest that it would be easy to select members for the Standards Committee, who, subject to the approval of the above bodies, would act as their representatives.

The Analytical Standards Committee should at the outset arrange :

1. For the provision of certain chemicals, such as benzoic acid, sodium oxalate, and sugar.

2. To report, after making full inquiries of manufacturers, users, and teachers, whether there is any legitimate scope for the extension of the scheme on the lines of the U.S.A. Bureau of Standards.

Further, the Standards Committee should ascertain to what extent and by what procedure the Department of Scientific and Industrial Research would support the Committee by a grant until the Committee's work becomes self-supporting.

The Council of the Institute have concurred with the proposals contained in the Report, and have nominated Mr. F. H. Carr, Dr. J. T. Dunn, Mr. Lewis Eynon, and Mr. F. W. Harbord, any of whom would be willing to act as representatives of the Institute in this connection.



REVIEWS.

APPLIED CHEMISTRY. By C. KENNETH TINKLER and HELEN MASTERS. Vol. i., 283 pp. and Index. Crosby, Lockwood, and Son. Price 12s. 6d.

Under this somewhat misleading though literally correct title Dr. Tinkler and Miss Masters have written the first volume of a work which is, we believe, unique alike in its intention and its scope.

It is an outcome of the establishment by the London University of a diploma in Household and Social Science. In view of the previous absence of any complete and definite scheme of instruction in the scientific aspects of domestic matters the importance of this new departure is obvious.

Until towards the end of the eighteenth century almost all the great country houses and many of the smaller ones were to a large extent self-supporting. Behind the scenes they were hives of industry; and kitchen and laundry, brewhouse and bakehouse, stillroom and spicery, loom and spinning-wheel, exacted from the housewives of the time wide and practical if not a scientific knowledge of what were then domestic arts and have now become matters of wholesale trade and manufacture.

The development of machinery, the applications of science to industry, and changed manners and customs now render us almost entirely dependent on commercial sources for the necessities as well as the luxuries of life. For the full understanding of domestic matters under the new conditions, knowledge of an entirely different kind is required, the acquirement of which has hitherto been by no means an easy matter.

The book before us deals with almost everything which could be reasonably included under the head of household science, food alone excepted, on which a further volume is to follow.

It is mainly intended as a guide to the *practical* work for the degree, matters of theory being left to the lectures except where necessary for the proper understanding of the experimental work.

As its title suggests, the major portion of the book is concerned with chemistry, treated chiefly but by no means exclusively from an analytical standpoint. Although they have already been dealt with in numerous textbooks, such subjects as the analysis of water, water softeners, soaps, dry-cleaning preparations, bleaching agents, disinfectants, air, coal gas, and liquid and solid fuels, together with the chemical and microscopical characters of the chief textile fibres, could hardly have been omitted from a work of this kind, but the authors have gone farther afield than this and have included in their syllabus the examination of paints, oils, and varnishes; of polishes for metal, wood, and leather; and of enamels and glazes for pottery and ironware.

The outstanding feature of the work, however, really lies in the attention devoted to subjects in which chemistry plays a minor part. Among these may be mentioned experiments in emulsification; in the measurement of surface tension (as an aid to the better understanding of the action of detergents); methods for determining the efficiency of gas stoves and geysers, also of the efficiency of the ventilation of rooms.

In fact, foods and building materials excepted, there is hardly a substance, process, or apparatus used in, on, or about a house which the authors have not taken within their purview; even the reading of gas meters and the explanation of the basis of the fuel equivalents in the now happily defunct Fuel Rationing Order receive due meed of attention.

The student who conscientiously does all the work described in this volume will have received a most valuable training in analytical and investigatory work. It is perhaps open to doubt whether such a mass of diverse subjects can be profitably dealt with in so brief a period as a single academic year; but apparently it is not intended that all the experiments, etc., described should be carried out by each student individually, some being included in the lectures and a few in the earlier years of their training.

This may to some extent justify what must otherwise be regarded as a defect in the book—namely, the inequality of the treatment of the various topics dealt with. Except for the purpose of teaching it to others, it is difficult to see exactly what use those who take the course for this degree will make of the knowledge they have acquired; but, whatever the answer to this problem, it can hardly justify the allocation of some thirty-two pages to a detailed description of the methods employed for the analysis of water, while only half that space is given to the examination of paints, varnishes, distempers, metal, furniture, and boot polishes, glazes and enamels, the analysis of which is often a matter of the utmost difficulty, and the published information about which is scanty and far to seek.

Taking the book as a whole it is most clearly and concisely written, and is remarkably free from errors, typographical or otherwise. It is very well printed, and most of the illustrations are excellent, except those of some of the ordinary pieces of apparatus (Figs. 1, 2, 3, and 4), which are very crude and in some instances quite out of proportion.

The authors may be congratulated on having written a most interesting work, which from its wide and novel range should appeal to many others besides those for whom it is specially intended.

CECIL H. CRIBB.

COURS DE CHIMIE. 2 vols. By R. DE FORCRAND. 1918-19. Paris: Gauthier-Villars et Cie. Second Edition. Price: Vol. I., 14 francs; Vol. II., 18 francs.

Professor Forcrand's book is written primarily for the guidance of French students preparing for the Certificat d'études supérieures de physique, chimie et sciences naturelles. The syllabus for this certificate comprises "the whole of chemistry," and the necessary instruction is to be conveyed, in eighty to ninety hours, to students who may begin with little or no chemical knowledge. While we must

admire the skill with which Professor Forcrand covers this comprehensive syllabus in a thousand pages, we must doubt whether his book can be of great service to those more fortunate students whose first year of study does not include "the whole of chemistry."

The book is divided into five sections dealing successively with General and Physical Chemistry, Inorganic Chemistry, Organic Chemistry, Analytical Chemistry, and Chemical Arithmetic. The section on Analytical Chemistry is necessarily restricted by the nature of the book. It suffers badly from the comprehensive programme; within one hundred pages the author deals with qualitative and quantitative inorganic and organic analysis, water analysis, and gas analysis. The section contains a good exposition of the general principles of analysis, but a few only of the detailed methods are given under each heading, and mention of these is often too brief to have much teaching value, either from the practical or theoretical standpoint. As it was not the intention of the author to write a guide to laboratory work, he might have made this section far more valuable by using his earlier chapters on theoretical chemistry to develop a more detailed system of one branch of analysis. Insufficient use has been made of the knowledge imparted in the first three sections. The statement (on page 332 of the second volume) that a test-tube is a thin, cylindrical glass tube, closed and rounded at one end, appears, for example, out of place; a student who has already been introduced to such subjects as the quinine and morphine alkaloids will know this.

The first section is the most valuable for the general reader. It contains an exceedingly clear and logically developed account of the fundamental chemical and physico-chemical principles. The section on inorganic chemistry is in need of further revision. The structural representation of the chlorine oxyacids with chains of oxygen atoms, and of hyposulphurous acid with two directly connected sulphur atoms, does not give the best graphical summary of the chemical properties of these compounds. In the same way, the statement that tannic acid is a digallic acid should have disappeared by now, even from the elementary textbook.

D. CLIBBENS.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

A STUDY OF THE DETERMINATION OF POTASSIUM AS THE PERCHLORATE, AND THE SEPARATION FROM SODIUM, ETC.

By R. LEITCH MORRIS, F.I.C.

(Read at the Meeting, June 2, 1920.)

THE work outlined below is a summary of nearly six years' experience of the perchlorate process, and it is claimed that, given attention to essential details, the results approach to scientific accuracy.

In the course of this work the strength of alcohol is expressed as percentage by volume. "Perchloric acid" means the aqueous acid, and in the experimental work a 20 per cent. solution was used. The formula HClO_4 refers to the absolute acid.

The insolubility of potassium perchlorate in strong alcohol was observed by Sérullas (*Ann. Chim. Phys.*, 1831, **46**, 294), who described an analytical process based on this fact.

Schloessing (*Compt. rend.*, 1871, **73**, 1269) seems to have been the first to publish trial results by the method. He evaporated the solution with perchloric acid nearly to dryness in a sand-bath, treated the cold residue with small quantities of strong alcohol, and decanted. "The more soda present the more sodium perchlorate is retained by the perchlorate of potassium; therefore it is best to dissolve the partly washed perchlorate of potassium in the least possible amount of hot water, and again evaporate to dryness. Two more washings with alcohol complete the washing."

Results given on a mixture of $\text{NaCl} + \text{KCl}$ are remarkably close to the truth. No determination on a pure potassium salt alone was made. The accuracy of these results may be ascribed partly to the use of nearly the minimum amount of alcohol (20 to 40 c.c. in all), and partly to the fact that the presence of sodium perchlorate considerably reduces the slight solubility of the potassium perchlorate in alcohol; in addition, the potassium perchlorate retains slight traces of the sodium salt even after the perchlorate residue remaining after the preliminary washing with alcohol is redissolved and the solution is again evaporated to dryness.

Wense (*Zeitsch. angew. Chem.*, 1891, 4, 691; 1892, 5, 233) used 96 per cent. alcohol containing 0.2 per cent. by weight of HClO_4 as the wash liquid, which considerably reduces the solubility of potassium perchlorate. The perchlorate residue was extracted twice with 10 c.c. of this mixture, and then collected and washed with the same liquid. Then, finally, it was washed with a few c.c. of pure alcohol to free it from traces of adhering perchloric acid. Tared filter papers were used. Drying temperature, 120° to 130° C. "The whole process requires 50 to 75 grms. of alcohol."

Caspari (*Zeitsch. angew. Chem.*, 1893, 6, 68) applied the process to the direct estimation of potash in fertilisers, ashes, etc., containing Fe, Mn, Al, P_2O_5 and alkaline earths. He recommended: (a) Filter tubes with asbestos; (b) wash liquid containing 97 per cent. alcohol and 0.2 per cent. HClO_4 ; (c) two evaporations with perchloric acid, finally to a syrup before treating with wash liquid; (d) extraction of cooled residue with 20 c.c. washing fluid, decanting and repeating extraction with 20 c.c. more; (e) dissolving residue in hot water and re-evaporating, as suggested by Schloessing; (f) transferring with the washing fluid, and finally washing with a few c.c. of pure alcohol. Drying temperature, 130° to 150° C.

These recommendations are improvements, excepting the use of 20 c.c. of wash liquid instead of the 10 c.c. recommended by Wense for each of the two preliminary extractions. The usual descriptions of the perchlorate process follow Caspari as regards the amount of wash liquid for extraction, but ignore nearly all the other details emphasised by him.

D. A. Kreider (*Amer. J. of Science*, 1895, 49, 433; reprinted, *Chem. News*, 1896, 73, 9-17) followed Caspari's details, using pure potassium chloride (0.1 grm.) and also a mixture containing a known amount of potassium chloride. A Gooch crucible is recommended. The results were good. In the case of the pure salt the error was $\frac{1}{10}$ to $\frac{4}{10}$ mgrm. below truth. Total filtrate, close to 50 c.c.

W. A. Davis (*J. Agric. Science*, 1912, 5, 52), made a detailed study of the process. He evaporated the solution once, on a sand-bath, with perchloric acid until it fumed strongly. The perchlorate residue was treated with 95 per cent. alcohol, and after decantation transferred and washed with alcohol to which 0.2 per cent. HClO_4 had been added, and finally washed with about 5 c.c. of pure 95 per cent. alcohol and dried at 100° C. The results are fair, but show somewhat wide variations. At the close of the paper he recommends that, after preliminary extraction with the 20 c.c. alcohol, the rest of the washing should be carried out with a solution of potassium perchlorate in 95 per cent. alcohol, saturated at room temperature.

Thin and Cumming (*J. Chem. Soc.*, 1915, 107, 361) recommend the use of the saturated solution of potassium perchlorate in 95 per cent. alcohol for the washing liquid as suggested by Davis. Beyond stating that the results are accurate they give no analytical data. Their paper is chiefly useful for their solubility determinations of potassium perchlorate in alcohol of various strengths, and in alcohol containing varying amounts of perchloric acid.

Baxter and Kobayashi (*J. Amer. Chem. Soc.*, 1917, 39, 249) have shown that absolute alcohol containing 0.1 per cent. HClO_4 dissolves at 0° C. as little potassium perchlorate as any practicable mixture (1.9 mgrm. per 100 c.c.), although the salt is still appreciably soluble even in this liquid. They recommend washing at a

temperature near 0° C. with a saturated (at 0° C.) solution of potassium perchlorate in the above liquid. They used a platinum sponge crucible and dried at 200° C. in an electric oven. The results were good, but working at 0° C. is obviously inconvenient.

In a later paper (*J. Amer. Chem. Soc.*, 1920, **42**, 735) they agree with the criticism of Gooch and Blake (see next paragraph) that potassium perchlorate may be precipitated from this washing solution when much sodium is present. They modify the process by making a preliminary extraction with 20 c.c. of absolute alcohol, containing 0.1 per cent. HClO_4 , but no perchlorate, and then washing with the perchlorate solution (saturated at 0° C.) already described. Otherwise, the process in detail is the same. A series of good results is quoted. In the presence of much sodium, re-solution of the precipitate in water, and re-evaporation at least once, is necessary.

Gooch and Blake (*Amer. J. Science*, 1917, **44**, 381) have shown that, at ordinary temperatures, the addition of a wash solution saturated with potassium perchlorate, to the perchlorate residue leads to precipitation of some of the dissolved potassium perchlorate, when much sodium perchlorate is present. (Cf. Baxter and Kobayashi.) They recommend the use of 97 per cent. alcohol containing 0.1 per cent. HClO_4 as wash liquid throughout the process, and avoid loss, through solubility of the KClO_4 , by restricting the amount of wash liquid used. They treat the perchlorate residue first with 10 c.c. only of washing fluid; decant; redissolve the residue and again evaporate when much sodium is present; treat again with 10 c.c., decant, and finally wash with 5 c.c. of the same wash liquid added in portions. In all their determinations the total filtrate was kept at 25 c.c. Repeated evaporation with perchloric acid is desirable if more than 0.2 gm. of $\text{NaCl} + \text{KCl}$ is present. (It will be seen that the final washing with pure alcohol usually prescribed is discarded; this washing with pure alcohol is the chief cause of the low results generally found in the perchlorate method.) The drying temperature is 130° C. If these details are followed, the problem of getting consistently accurate results by the perchlorate process is solved without the use of a saturated perchlorate solution for the washing.

The process I have adopted since 1914 is the same in nearly every detail as Gooch and Blake's.

The perchlorate process has also been applied by some workers to the precipitate of potassium-sodium-cobaltinitrite. This is a useful process when sulphates or tartaric and citric acids are present. Bennett (*ANALYST*, 1916, **41**, 165) used it. The results are good; generally a little higher than truth.

Notes on the Varying Procedures given above.—Wense's suggestion of using 10 c.c. only, of the wash liquid for each washing by decantation has been neglected. I had adopted it before I saw Gooch and Blake's paper. Most text-books follow Caspari's quantities of 20 c.c. each time. This increased amount is probably of little moment in the case for which Caspari describes the method—viz., the direct determination of potash in mixed fertilisers, etc., where considerable amounts of other bases and also phosphoric acid may be present; but when only a pure potassium salt is present, low results will be obtained. The final washing with pure alcohol to remove traces of perchloric acid is unnecessary when asbestos filters are used, and

the precipitate dried at 130° C. or higher. Davis dried at 100° C.; a higher temperature is better.

The wash liquid should contain at least 97 per cent. alcohol after the addition of the perchloric acid, and there is no objection to using absolute alcohol containing perchloric acid. With lower alcohol strengths than 97 per cent. the solubility of the potassium perchlorate increases considerably.

Two evaporations with perchloric acid should be made; if an excess of perchloric is present at the end of the first evaporation, the second evaporation will suffice in all cases. The Gooch crucible is the best filter. At least one solution of the precipitate followed by re-evaporation is necessary, after preliminary extraction with wash liquid, when sodium or other bases are present; otherwise the results may be above the truth. Finally, it is advisable to keep the total amount of wash liquid as low as possible, as too free use of it will make the results low.

Influence of other Substances present: Ammonium Salts must be absent.—The results obtained by Schloessing show that the presence of alkaline earths and magnesia exerts but little effect on the results, and Caspari has shown that it is not necessary to remove phosphoric acid, and that it is only necessary to remove sulphuric acid. This last is absolutely essential, owing to the insolubility of sodium sulphate in alcohol. In his paper Davis shows that fair results are obtained on pure potassium sulphate without removing the sulphuric acid, and states that it is not always necessary to remove sulphuric acid; but he seems completely to have overlooked the point that the removal of sulphuric acid is necessitated in general analysis by the insolubility of sodium sulphate in alcohol, and he himself found that, when magnesium was present as sulphate, the removal of the sulphuric acid was necessary. In a later paper (*J. Chem. Soc.*, 1915, 107, 1680) he recommended the removal of sulphuric acid in all cases except in the analysis of pure potassium sulphate. It is evident that Davis assumed that sodium sulphate on evaporation with perchloric acid behaves similarly to potassium sulphate. This is not the case; the perchloric acid treatment leaves sodium sulphate unchanged. This is a very important point to note, and is referred to below, under "Experimental."

As regards magnesium, Kreider treated 0.15 grm. magnesium carbonate with excess of perchloric acid, evaporated the solution, and treated the cold residue with alcohol, when a perfectly clear solution was obtained.

Practically all perchlorates seem to be freely soluble in strong alcohol except those of potassium, rubidium, caesium, and ammonium. Ammonium perchlorate is slightly soluble in alcohol—about 2 per cent. In using the perchloric process, it is essential to use sufficient perchloric acid to convert completely all the bases present into perchlorates, owing to the insolubility of most chlorides, etc., in alcohol. Kreider found that, to secure a nearly complete separation of phosphoric acid from the potassium, a considerable excess of the perchloric acid should be left upon the potassium perchlorate before it is treated with alcohol.

Textbook Descriptions of the Process.—These, in general, are faulty. Mellor's "Treatise" follows Caspari generally, but omits the re-dissolving and re-evaporation of the residue after the first treatment with the alcoholic fluid. The final washing with pure alcohol is omitted; this is an improvement.

Treadwell (*Quant. Anal. II.*, English edition) describes the process accurately in Caspari's own words, but makes a serious error at the end by saying that, finally, the precipitate is to be washed with 50 to 75 c.c. of pure alcohol. If this is done, the results must be low, unless in the case where a very large amount of sodium is present. This error has arisen through misreading Wense's words, "the whole process requiring 50 to 75 grms. of alcohol," this implying a total filtrate of 60 to 80 c.c. There seems little doubt that this description may have led to the condemnation of the process by some workers who have tried it on a pure potassium salt, following Treadwell's directions, and found the results too low.

The Stassfurt modification of the perchlorate process for crude potassium salts is described by Lunge and Keane (*Tech. Methods of Chem. Anal.*, Vol. I., Part II., p. 530). The details resemble those of Caspari, but the re-dissolving of the perchlorate is neglected. The washing solution is 96 per cent. alcohol, to which 2 per cent. (of 20 per cent.) perchloric acid has been added. Finally, the perchloric acid is removed by washing with a minimum of pure 96 per cent. alcohol. A water-bath is specified for the evaporation; this is unsatisfactory, a higher temperature (120° to 130° C.) is necessary for the final stage of the evaporation, since the perchloric acid does not reach the fuming-point at the temperature of the water-bath. The washing solution, it will be noted, contains only 94 per cent. alcohol and 0.4 per cent. HClO_4 ; 0.2 per cent. HClO_4 is sufficient; the extra amount of acid reduces the alcohol content by an extra 1 per cent.

Perchloric Acid.—Pure perchloric acid, "reagent," has only been obtainable commercially in the last fifteen years. Kreider described a very convenient method of preparing the acid from pure sodium perchlorate by treating it with fuming hydrochloric acid (sp. gr. 1.2), filtering off the sodium chloride precipitated, on asbestos, and concentrating the filtrate to the fuming-point. Such an acid contains about 40 to 50 mgrms. of sodium perchlorate in 1 c.c., and is of 60 per cent. by weight (equal to nearly 1 gm. HClO_4 per c.c.).

Thin and Cumming found that Merck's "pure" perchloric acid (20 per cent.) gave 0.1476 gm. solid residue from 10 c.c. and contained some potassium perchlorate. Another lot of acid gave 0.049 gm. from 10 c.c.

Perchloric acid of reagent purity has been obtainable both from a German, and at least one English source since 1910, such acid leaving only traces of visible matter on evaporation of 10 c.c. and giving no result for a blank when put through the analytical process for potash.

STRENGTHS AND SPECIFIC GRAVITIES OF PERCHLORIC ACID SOLUTIONS.

Specific Gravity.	HClO_4 (Per Cent. by Weight).	Boiling-Point.	HClO_4 grms. per 100 c.c.
1.782 at 15.5° (Roscoe)	100	110° (Roscoe)	
*1.82 (Roscoe)	71.6	203° (Roscoe)	120
1.12	20	—	22

* Aqueous acid of constant boiling-point, approximating to the composition of the dihydrate $\text{HClO}_4 \cdot 2\text{H}_2\text{O}$.

I have found the commercial "pure" 20 per cent. acid free from potash, but a blank analysis gives 1 to 2 mgrms. of residue (per 10 c.c. of the acid) insoluble in alcohol, but this is nearly always found to be silica only. Davis apparently used such an impure acid and did not correct for it in his results.

It is noticeable that, according to Roscoe, the pure acid has a much lower sp. gr. and also a lower boiling-point than the 71 per cent. acid. (*J. Chem. Soc.*, 1863, 16, 82.) From the content of HClO_4 by weight in volume, it is evident that the 70 per cent. acid is nearly five and a half times stronger in HClO_4 than the 20 per cent. acid, when equal volumes are taken. Later workers have used the 20 per cent. acid, while the acid of Kreider, and also of Caspari, was of about 60 per cent. Wash-alcohol, containing 0.2 per cent. HClO_4 , is 1 per cent. weaker in alcohol (when the 20 per cent. acid is used) than the pure alcohol used for the preparation. Kreider has also described the preparation of the purified acid (70 per cent.) by distillation, under reduced pressure. Gooch and Blake in their work used acid prepared in this way of 70 per cent.

Experimental.—In 1914 I had occasion to determine the potash in a mineral water of the Apollinaris type, and used the perchlorate process for this, following the procedure described below. Some further preliminary work showed that the process was accurate, and that it was unnecessary to wash with a solution of perchlorate of potassium in alcohol. The process was also used, in general practice, to check the amount of potash salt in solutions prepared on a manufacturing scale.

Fresenius (*Quant. Anal.*) recommended the use of the filtrate to transfer potassium platinichloride and also magnesium ammonium arsenate to the filter, in order to avoid over-washing. I adopted this procedure for the transference of the perchlorate, to avoid using more than 30 to 40 c.c. of washing fluid in all. The trial analyses were undertaken to find accurately the experimental error of the method, using varied amounts of potassium salt. The publication of Gooch and Blake's paper (*loc. cit.*) confirmed my own results, but the trial-analyses were continued to supplement their work. A few analyses using increased quantities of wash liquid were also made in order to compare the results. A total of 25 c.c. of wash liquid is not always quite sufficient to wash the precipitate thoroughly when considerable amounts of sodium or other salts are present.

Reagents.—The perchloric acid used was a 20 per cent. solution (analytical reagent). Ten c.c. of the acid left, on evaporation and drying at 140°C ., usually about 3 mgrms. of residue. This residue, however, when treated with the alcoholic wash liquid, usually left nothing weighable on the filter. More recently I have found a residue, after filtering, amounting to 0.4 mgrm. per 10 c.c. acid. The trace of insoluble residue is apparently silica. In using this batch of acid, the corresponding correction has been made on the perchlorate obtained in the analysis. The potassium chloride used was Kahlbaum's "K" brand, "for analysis."

Wash Liquid.—Made as follows :

Alcohol, 98 per cent.	...	...	...	100 c.c.
Perchloric acid	...	...	...	1 c.c.

This wash liquid will have a content of 97 per cent. alcohol, and will contain, practically, 0.2 per cent. HClO_4 .

The 98 per cent. alcohol can be easily made by mixing 110 c.c. of absolute alcohol (of not less than 99.5 per cent.) with 100 c.c. of 96 per cent. alcohol.

Process.—In nearly all cases the solution of the salt was evaporated with perchloric acid, three times in all, and in the final evaporation taken practically to dryness. The residue (in the analyses following after No. 8 and No. 3A of Tables I. and II.) was treated with 10 c.c. of wash liquid, stirred well with a flat-headed glass rod, allowed to stand ten to fifteen minutes when sodium was present, occasionally stirring; the solution allowed to settle, and the liquid decanted through a weighed Gooch crucible containing asbestos. When only potassium was present, the precipitate was then rinsed out directly with the wash liquid, in measured quantity, by means of a wash-bottle. When sodium was present the basin containing the residue from the first decantation was placed on the top of the air-oven for a few minutes, till the alcohol was evaporated; then the residue was dissolved in the minimum amount of boiling water, and again evaporated to dryness. The treatment with 10 c.c. of wash liquid and decantation was repeated, and the residue then transferred completely by using the latter 10 c.c. of the filtrate. Finally, a measured volume of wash liquid was added (by the use of a small pipette) in portions, to complete the washing on the filter. The Gooch crucible and contents were then dried for about an hour at 130° to 150° C., cooled for one hour in a desiccator and weighed. After weighing, 2 to 3 c.c. more wash liquid were passed through the filter, and the drying and weighing repeated. The weight was taken as constant when, on washing with 2 to 3 c.c. of liquid, the difference on successive weighings did not vary by more than one or two tenths of a milligram.

In all cases the weighings were made by the method of vibrations; pipettes and weights were calibrated, and the solutions used were adjusted at 15° C., and measured off after being always brought to the same temperature. The usual variations obtained in weighing crucibles, etc., which may easily amount to 0.0003 gm. or more, due to absorption of air and moisture by the glaze of the crucible during weighing, were thus minimised by weighing quickly. On the average, the weighings may be taken as correct within ± 0.0001 gm.

Apparatus, etc.—In nearly all cases porcelain Gooch crucibles of 10 c.c. capacity were used; in one or two instances only, a 20 c.c. quartz "Gooch" was employed. For amounts of potassium perchlorate from 0.4 gm. upwards the 10 c.c. "Gooch" is rather small. The same asbestos felt was used over and over again, the crucible, after finishing an analysis, being washed out repeatedly with boiling acidulated water. After six or eight estimations, the interior of the crucible will be found to be slightly stained yellowish brown, and the weight of the empty crucible will show an increase of 0.1 to 0.2 mgrm., even though the acid gives no weighable residue in the blank process. This colour is apparently due to the action of perchloric acid on the alcohol or on the impurities (*i.e.*, the traces of aldehydes or esters) contained in the alcohol. In practice it is well to ignite the empty crucible containing the asbestos after six determinations have been carried out; then to pass some distilled water through the crucible, and dry at 130° to 150° C. as usual.

It is essential to have proper facilities for drawing off the filtrate conveniently. The usual pressure-flask is quite inconvenient. Filtering direct into a beaker or

flask inside an exhausted bell-jar is satisfactory, but the handiest apparatus is the Gooch filter-tube, No. 1180 in Townson and Mercer's list. This is a wide-mouthed cylindrical separating funnel, with stop-cock at bottom, and a side tube to connect with the pump.

The Wash-Bottle.—It is best to use a wide test-tube for this, as the small amount of wash liquid used makes even a small flask unsuitable. The outlet tube and jet should be made from one piece of glass tubing, as contact with rubber connections is best avoided. The tip of the jet should be drawn out very finely. The portions of wash liquid used should be approximately measured.

It is best to use the wash-bottle in transferring the filtrate, rinsing out directly into the "Gooch"; after nearly all the precipitate appears to be transferred, it is essential to rub round carefully the sides of the dish, by using the tip of a feather as a brush. It is very difficult to know when the dish has been properly cleaned, the alcohol seems to reduce the visibility of the precipitate and the usual rubber-tipped rod is unsuitable for this transference, as the rubber soon gets slightly sticky, owing to the action of the trace of perchloric acid in the washing fluid.

Platinum or quartz dishes are preferable to glass. A white porcelain basin is useless. A blue glazed porcelain basin (Royal Worcester ware) was used in some cases; this is satisfactory for enabling one to judge when all the precipitate has been transferred. But Gooch and Blake have noticed that perchloric acid has a decided action on glass, and the results in the Tables I. and II. seem to show a similar action in the case of the blue glazed porcelain. In such cases the perchlorate of potassium may contain silica.

Evaporation.—A water-bath is unsuitable, since the perchloric acid cannot be concentrated to the fuming-point below 120° C. Davis recommended a sand-bath, which is inconvenient in quantitative work. It is best to use some form of hot air-bath. The simplest device is to use a shallow sand-bath tray, covered with a set of rings, on which the dish is set. The heat is applied by means of a burner with a rose top. With a little practice the temperature is easily adjusted to allow of speedy evaporation, without the liquor ever boiling, even towards the end. In all these test analyses, the air-bath and the dish were placed under an inverted Victor Meyer funnel, so as to exclude dust from the atmosphere.

The following hints on preparing the asbestos felt in the "Gooch" may be useful: Shake up a fair quantity of the prepared asbestos emulsion in water, let the whole settle partially by leaving it to stand two or three minutes. Decant off the liquid containing the finer particles of asbestos into another bottle. Prepare the "Gooch" by running through some of the coarser emulsion, till the perforations are evenly covered. Then add a few c.c. of the finer emulsion to fill up the larger interstices that may be left in the first layer of the felt. Then put in a perforated plate, and add a little more asbestos as before. Such a felt will retain the perchlorate in all cases, even though the previous stirring may have reduced the precipitate to fine powder. With similar precautions, an asbestos felt can be prepared to retain calcium oxalate, and even barium sulphate, and yet to filter quickly under a medium pressure. With this device, a very thin layer of asbestos suffices.

It is sometimes advised to avoid breaking up the precipitate too much, in case it

becomes too fine to be retained by the filter. The writer's opinion is that it is best to stir up thoroughly and grind to powder by the flat-headed rod. In none of these experiments was a cloudy filtrate ever obtained with potassium perchlorate. In one case the filtrate was slightly opalescent, but this was due to a trace of barium sulphate being present in the perchlorate.

In the case of a direct determination of potash in presence of barium, it is advisable to use a large asbestos card, with a hole in the centre, as the support for the dish instead of the rings. This will partly, at least, deflect the products of combustion of the gas away from the dish, and so help to prevent the formation of any considerable quantity of barium sulphate.

TABLE I.—POTASSIUM ONLY PRESENT.

No.	Potassium Chloride taken (Grm.).	Potassium Perchlorate obtained (Grm.).	Vessel.	Temperature (C.).	Volume of Filtrate (c.c.).	KCl found (Grm.).	Procedure.	Potash Salt recovered (per Cent.).
1	0.2456	0.4551	Glass	13°	60	0.2449	20 c.c. + 20 c.c. + washings	
2	0.1368	0.2544	"	13°	60	0.1369		
3	0.1368	0.2539	"	13°	60	0.1366		
4	0.1368	0.2544	Quartz	10°	25	0.1369	10 c.c. decanted and precipitate rinsed out by wash-bottle Filtrate occasionally used to transfer the precipitate	100.07 100.09 99.90 100.18 99.60 99.90 Average 99.96
5*	0.3249	0.6043	"	10°	35	0.3252		
6*	0.3935	0.7305	"	10°	35	0.3931		
7*	0.6673	1.2424	Glass	10°	35	0.6685		
8	0.0988	0.1829	Quartz	21°	27	0.0984		
9	0.0988	0.1834	"	23°	25	0.0987		
10	0.0273	0.0505	"	5°	25	0.0272		
11	0.0131	0.0242	"	15°	25	0.0130		
12	0.0122	0.0230	"	13°	25	0.0124		
13	0.0122	0.0229	"	13°	25	0.0123		
14	0.0122	0.0227	"	13°	25	0.0122		
15	0.0122	0.0230	Blue glazed porcelain	13°	25	0.0124		
16	0.0152	0.0281	Quartz	21°	25	0.0151		
17	0.0152	0.0282	"	24°	25	0.0152		
18	0.0061	0.0113	"	15°	25	0.0061		
19	0.0062	0.0115	"	15°	27	0.0062		
20	0.00304	0.0055	"	15°	25	0.00296		
21	0.00306	0.0055	"	15°	25	0.00296		

The arrangement of results is in descending order of magnitude, according to the amount of KCl taken, and the results can be directly compared with the corresponding numbers of Table II. The usual order of working was to make two or more analyses of each solution of potassium chloride made up, then, taking the same amount of solution, to add the NaCl as shown in Table II.

* KCl in Nos. 5, 6, 7 weighed off separately.

TABLE II.—POTASSIUM IN PRESENCE OF SODIUM.

No.	KCl taken (Grm.).	NaCl taken (Grm.).	KClO ₄ weighed (Grm.).	Temperature (C.).	Volume of Filtrate (c.c.).	KCl found (Grm.).	Treatment of Perchlorate Residue.*	Potash recovered (per Cent.).
2A	0.1368	0.13	0.2525	13	100	0.1359	} 20 c.c. + 20 c.c. + wash- ings. Not redissolved	
3A	0.1368	0.20	0.2547	13	90	0.1371		
4A	0.1368	0.2	0.2562	5	30	0.1379	10 c.c. + 10 c.c. + wash- ings. Not redissolved	100.22 100.20 100.14 100.10 99.8 } Average 100.09
4A 1	0.1368	0.2	0.2547	5	30	0.1371	Redissolved	
5A †	0.3511	0.3	0.6538	10	35	0.3518	"	
5A 1 †	0.2083	0.5	0.3877	10	35	0.2086	"	
8A †	0.0988	0.2	0.1838	20	27	0.0989	"	
9A	0.0988	0.2	0.1833	21	28	0.0986	"	
10A	0.0273	0.2	0.0512	10	32	0.0276	Not redissolved	
10A 1	0.0273	0.2	0.0502	10	30	0.0270	Redissolved	
10A 2,	0.0273	0.2	0.0505	10	32	0.0272	"	
12A	0.0122	0.5	0.0242	15	30	0.0130	Not redissolved	
13A	0.0122	0.3	0.0236	15	33	0.0127	Redissolved	
14A	0.0122	0.3	0.0234	15	30	0.0126	"	
15A	0.0122	0.2	0.0221	15	30	0.0119	"	
18A	0.00611	0.12	0.0114	15	30	0.00613	"	
20A	0.00305	0.13	0.0056	15	27	0.00301	Not redissolved	
22§	0.4590	0.5	0.8622	15	35	0.4639	Not redissolved	
23§	0.5096	0.5	0.9618	15	35	0.5175	" "	
24	—	0.5	0.0001	15	35	0.0000		

* 4A to 23 inclusive—10 c.c. + 10 c.c. + washings. Transferred by filtrate from second 10 c.c.

† KCl weighed off separately.

‡ Through not having a "Gooch" crucible prepared, the perchlorate, after treatment with 10 c.c. of wash liquid, stood all night before filtration. Glass dish used for 2A and 3A. Green glazed dish for 5A. Quartz dish for all others. The term "redissolved" means that the residue after the first decantation was dissolved in hot water and again evaporated to dryness.

§ In Nos. 22 and 23 the errors on KCl are respectively: +0.0049 and +0.0079. This shows the necessity of redissolving the residue as described, in the case of much sodium being present. Baxter and Kobayashi found a somewhat similar error.

In Table III. the KClO₄ was redissolved and the solution again evaporated (after the first decantation), $\frac{1}{2}$ to 1 c.c. more perchloric being added. The precipitate was transferred by the second portion of the filtrate.

A. Taken to dryness, treated with 10 c.c. of washing fluid, decanted. After the re-evaporation, 10 c.c. of washing fluid again used and decanted.

B. Taken to a moist pastiness, 10 c.c. of absolute alcohol added and decanted; 10 c.c. again added to the pasty residue on re-evaporation with addition of a little perchloric acid, and the decantation repeated.

C. Taken to dryness, 0.2 c.c. perchloric acid added, well stirred, then 10 c.c. of 98 per cent. alcohol added, and this treatment repeated. Results good.

D. Same as C, except that 12 c.c. of absolute alcohol used each time in place of 98 per cent. Results too high.

E. Same as C, but using double the amount of perchloric acid, 0.4 c.c. each time.

F. Same as B, using 15 c.c. of 98 per cent. alcohol for each decantation.

TABLE III.—POTASSIUM IN PRESENCE OF SODIUM PHOSPHATE.

No.	KCl taken (Grm.).	Na ₂ HPO ₄ ·12H ₂ O (Grm.).	KClO ₄ Weighed (Grm.).	Temperature (C.).	Volume of Filtrate (c.c.)	KCl found (Grm.)	Error on KCl (Grm.).	Process (see above).	Potassium Salt recovered (per Cent.).
1	0.0988	0.4	0.1878	15	35	0.1010	+ 0.0022	A	
2	0.0988	0.26	0.1834	20	29	0.0987	- 0.0001	B	
3	0.0988	0.4	0.1842	20	40	0.0991	+ 0.0003	B	
4	0.0988	0.4	0.1828	18	30	0.0984	- 0.0004	B	
5	0.0988	0.4	0.1838	16	26	0.0989	+ 0.0001	C	
6	0.0988	0.4	0.1834	16	30	0.0987	- 0.0001	C	
7	0.3447	1	0.6613	15	45	0.3558	+ 0.0111	D	
8	0.3447	1	0.6617	15	45	0.3560	+ 0.0113	D	
9	0.3447	1	0.6436	13	46	0.3463	+ 0.0016	E	
10	0.3447	1	0.6397	13	46	0.3442	- 0.0005	F	99.86
11	0.3447	1	0.6420	12	45	0.3455	+ 0.0008	F	100.24
12	0.3447	1	0.6408	10	49	0.3448	+ 0.0001	F	100.01
									Average 100.03

NOTES TO TABLE III.—In presence of phosphoric acid it is evidently unsafe to evaporate the perchloric solution to complete dryness, as the phosphoric acid, if nearly completely dehydrated, dissolves very slowly in the wash liquid, and cannot be completely washed out of the precipitate in such cases. (See No. 1, Table III.) Kreider recommends in this case that a considerable excess of perchloric acid should be left in contact with the residue before treatment with alcohol. Caspari, in describing his process for the case of fertilisers, etc., where phosphoric acid and alkaline earths are present, evaporated the solution with perchloric acid to "syrupiness" only, and recommends the avoidance of evaporating off too much perchloric acid. This means that varying amounts of water (in the perchloric acid) will be left on the residue, and hence, on treatment with the washing fluid, the alcoholic strength of the latter will be considerably reduced by an indefinite amount, varying with the excess of perchloric acid left on the residue.

For this reason the expedient was tried of taking the residue first to dryness, and then moistening it with 0.1 or 0.2 c.c. of perchloric acid, and adding 10 c.c. of pure strong alcohol of 98 per cent., so as to keep the alcoholic strength of the liquid in contact with the precipitate as near 97 per cent. as possible. (Procedure C.) Final washings were made with the usual mixture.

This procedure gives good results (Nos. 5 and 6, Table III.), but when tried on

larger quantities of sodium phosphate the results were considerably too high (see Nos. 7 and 8, Table III.). Process F, however, gives good results. It seems best, then, to evaporate to a moist pasty condition when much phosphoric acid is present, and to treat the residue with 15 to 20 c.c. of 98 to 100 per cent. alcohol for each decantation. Finally to wash with the usual wash liquid. The free phosphoric acid present seems to have no effect on the solubility of the potassium perchlorate in the washing fluid.

Determination in Presence of Magnesium :

1.	Taken	0.0988	gram.	KCl + 0.07	gram.	MgO	...	...	Found	0.1050	gram.	KCl.
2.	"	0.0988	"	KCl + 0.2	"	MgO	...	...	"	0.0990	"	KCl.
3.	"	0.0988	"	KCl + 0.2	"	MgO	...	...	"	0.0990	"	KCl.

Filtrate in each case—32 c.c. First residue in each case redissolved after first decantation with wash liquid.

The result of No. 1 is too high. On washing with 5 to 10 c.c. more wash liquid the loss was but slight, and the perchlorate still showed the presence of magnesium. (This is probably due to some over-heating in trying to carry the evaporation to dryness. When fumes of perchloric acid had nearly ceased coming off, the residue was still pasty while hot, and the temperature was carefully raised to get nearly complete dehydration. It is probable that magnesium perchlorate, which seems to crystallise with a considerable amount of water of crystallisation, partially decomposes before complete dehydration can be effected, some magnesium oxide being formed. Magnesium chloride behaves in this manner, and, apparently, the perchlorate of magnesium resembles the chloride in this respect. On account of this behaviour, in Nos. 2 and 3 above, the evaporation was stopped when the fumes of perchloric acid were still just faintly visible. The pasty residue so obtained sets solid when removed from the heat, and, when cold, forms a hard cement-like mass. It is best to add the 10 c.c. of wash liquid to this, and to set aside for an hour or two before attempting to break up the mass. Any premature attempt at stirring up the mass will inevitably lead to loss by splashing. It is evident from the note on Experiment 1 above, that the washing must be complete, since passing more wash-liquid through the Gooch, after drying at 130° to 150° C., will have but little effect in removing any magnesium left on the perchlorate, as the magnesium will, after the drying, be partly present as magnesium oxide.

Separation from Calcium.—Calcium, evidently, does not interfere with the process. In the evaporation the mass of perchlorates obtained (which is semi-fluid while hot, although fumes of perchloric acid have almost ceased) sets to a hard mass, and the disintegration of this by the wash liquid is best carried out as described under magnesium.

Taken	0.0988	gram.	KCl + 0.07	gram.	CaCO ₃	...	Found	0.0986	gram.	KCl	Filtrate
"	0.0988	"	KCl + 0.14	"	CaCO ₃	...	"	0.0990	"	KCl	30 c.c.

In Presence of Calcium and Magnesium together.—Taken 0.0988 gram. KCl + 0.07 gram. CaCO₃ + 0.07 gram. MgO. Found 0.0985 gram. KCl. Filtrate, 30 c.c.

In Presence of Barium.—Barium *per se* does not affect the results, but the con-

version into perchlorate must be complete, since barium chloride is very insoluble in alcohol; still, in practice, for accurate results, it may be advisable to remove the barium when considerable quantities are present, unless the evaporation can be carried out on some form of electrically heated radiator; otherwise, the potassium perchlorate may contain appreciable amounts of barium sulphate, owing to the gas. At a very early stage of these test analyses, the influence of barium was investigated, but the experiment failed, owing to this cause. After the first evaporation, on adding water to redissolve, the residue gave a cloudy solution, which was due to the formation of barium sulphate.

In the results below, the evaporation was carried out on the air-bath described, using a large asbestos card to deflect the products of the burnt gas away from the liquid. Even then, traces of barium sulphate were present in the perchlorate weighed.

1. Taken 0.0122 grm. $\text{KCl} + 0.2 \text{ BaCl}_2 \cdot 2\text{H}_2\text{O}$. Found at first 0.0126 grm. KCl . Corrected by deducting BaSO_4 present, 0.0123 grm. KCl .

2. Taken 0.0062 grm. $\text{KCl} + 0.1 \text{ BaCl}_2$ cryst. $+ 0.2 \text{ MgCl}_2$ cryst. $+ 0.05 \text{ CaCl}_2$ cryst. Found 0.0057 grm. KCl . In this last case the filtrate was slightly opalescent, probably a trace of BaSO_4 was present, which passed through the asbestos.

Determination of pure Potassium Sulphate.—Davis found that the direct estimation of pure potassium sulphate was possible, and the following analyses were therefore tried.

It is obvious that, on evaporating potassium sulphate solution with the perchloric acid, free sulphuric acid is liberated, and that the free sulphuric acid cannot be removed by evaporation without reversing the reaction. For this reason the evaporation was carried out at as low a temperature as would suffice to drive off the perchloric acid in fumes. This leaves the residue of perchlorate quite moist, since the free sulphuric acid at this temperature must still be in a fairly dilute condition. On now adding the wash liquid to this moist residue, a considerable reduction of the alcoholic strength will take place, and somewhat low results may be expected. In analysis 1 below, the ordinary wash liquid (10 c.c.) was used to pour on the residue and decanted, whilst in analysis 2 absolute alcohol (10 c.c. containing 0.2 per cent. HClO_4) was used, to compensate somewhat for the reduction in the strength of the alcohol, the remaining washings, etc., being carried out with the usual wash liquid:

1. Taken 0.1072 grm. K_2SO_4 . Found 0.1066 grm. Filtrate 25 c.c.

2. Taken 0.6704 grm. K_2SO_4 . Found 0.6664 grm. Filtrate 30 c.c.

It will be noted that these results are somewhat low for the reason given above.

Davis, using 0.100 grm. K_2SO_4 , recovered 99.1 per cent. in two cases.

Davis's results on a solution containing 0.04 grm. $\text{KCl} + 0.01$ grm. K_2SO_4 were fairly satisfactory, but in stating that it is not always necessary to remove sulphate, he completely overlooked the fact that this removal is necessitated by the insolubility of sodium sulphate in alcohol, and that sodium sulphate is not decomposed into sodium perchlorate by evaporating with perchloric acid. The reason for this difference in behaviour is easily found in Berthollet's law. Potassium perchlorate, being the more insoluble salt, is precipitated, while the opposite is the case with the sodium salt, sodium perchlorate being more soluble than the sulphate.

It is fairly evident, then, that if we take a mixture of potassium sulphate and sodium chloride, high results may be expected, owing to the sodium sulphate being formed. To prove actually by experiments that such is the case, the following analyses were carried out :

1. Taken 0.2627 grm. K_2SO_4 + 0.06 grm. NaCl. Found 0.3099 grm. K_2SO_4 . Error + 0.0472 grm.

2. Taken 0.2592 grm. K_2SO_4 + 0.05 grm. NaCl. Found 0.2975 grm. K_2SO_4 . Error + 0.0383 grm.

On calculating the NaCl taken into its equivalent of sodium sulphate, it was found that this figure came within a few milligrams of the difference between the potassium perchlorate actually weighed and the theoretical amount of perchlorate. It seems conclusive that the error shown in the results is due to the presence of normal sodium sulphate. It is important to notice that evaporation of a solution of sodium sulphate with perchloric acid leaves the sulphate of sodium quite unchanged.

In Presence of Ferric Salts.—Taken 0.3447 grm. KCl + 0.1 $FeCl_3$. Found 0.3445 grm. KCl. Filtrate 32 c.c.

The solution was evaporated with perchloric acid twice, and in the second evaporation was taken nearly to complete dryness, treated with 10 c.c. of wash liquid, decanted, and the perchlorate transferred as usual, without a previous re-crystallisation. It was noticed that the yellow colour of the solution gradually became weaker as the solution approached dryness, and that in the second evaporation the solution was colourless throughout. The residue obtained was pure white, but on treating with the wash liquid the strong yellow colour of the solution reappeared. After washing and drying the perchlorate was perfectly free from any yellow tint. It seems obvious, then, that no possible contamination of the perchlorate with ferric oxide need be feared.

Aluminium perchlorate, also, is perfectly soluble. It is possible, however, that any overheating of the dry residue might lead to high results in the case of iron and aluminium. But if the solution is evaporated as described above, there would seem to be no possibility of this occurring.

In Presence of Citric and Tartaric Acid.—Caspari stated that the process is suitable in such cases, and therefore some trials were made on a specially purified potassium hydrogen tartrate (free from traces of calcium and sulphates).

On carrying out the process as usual, it was found that, in trying to evaporate off the perchloric acid, the mass swelled and turned brownish-black. In another trial the solution was evaporated as far as possible on the water-bath, then transferred to the air-oven at a temperature of about 110° C. Even at that temperature the charring commenced. The following result was obtained by using a very slight excess of perchloric acid over the theoretical amount (0.5 c.c.) and the solution was evaporated, as far as possible, on the water-bath only. The residue was still semi-fluid; 10 c.c. of pure 98 per cent. alcohol were added, and decanted. The transference and washing were then carried out with the usual washing fluid. The results are low, as must be expected, since the perchloric acid cannot be concentrated very far on the water-bath.

In this case it would seem preferable to apply the cobaltinitrite process for the

precipitation of the potash, treating the precipitate then obtained with perchloric acid as usual, but no trial of this process was made.

Taken 0.1005 grm. $\text{KHC}_4\text{H}_4\text{O}_6$. Found 0.0994 grm. Filtrate 30 c.c.

Citric acid behaves similarly to tartaric acid in the evaporation.

Analysis of Samples supplied by Mr. Bertram Blount.—Early in 1919 a selection from the results of Tables I. and II. was sent to Mr. Blount, with the request that he would send a few samples on which to check the degree of accuracy obtainable by the procedure given. Mr. Blount kindly sent some samples. Nos. 1 and 2 were stated to be mixed chlorides of potassium and sodium.

Sample 1 was analysed as received; transferred from the sample tube to a weighing-bottle and the amounts taken weighed off by difference.

The following results were obtained:

	Samples Weighed off separately.	Equal Volumes of the same Solution used.
No. 1.	KCl per cent. 36.42, 36.13, 36.66.	36.14, 36.32, 36.11.
	Average of six results ...	36.30 per cent.
	By platinum process (Mr. Blount) ...	36.40 per cent.

As the above six individual results showed rather a wide divergence between the minimum and maximum found, the whole of sample 2 was rapidly mixed in a mortar before transferring to a weighing-bottle, and all the weighings-off were done immediately.

The results on Sample 2 were:

	Samples Weighed off separately.	Equal Volumes of same Solution used.
No. 2.	KCl per cent. 75.64, 75.38.	75.45, 75.54, 75.62.
	Average of five results ...	75.53 per cent. KCl.
	By platinum process (Mr. Blount)	75.53 per cent. KCl.

Series A, B, C, D. Determination of KCl and NaCl in a solution containing also chlorides of Al, Mg, and Ca.

This was done in order to show what results might be expected in the determination of the alkalis in a complex mixture, after removing all other bases, and isolating the mixed alkaline chlorides; and to compare results with a direct determination of the potash without previous removal of the other bases. Great care was taken to eliminate the Ca, Mg, etc., as completely as possible before weighing mixed chlorides, and, after the weighing, the chlorides were dissolved in a little water and any residue ignited, weighed, and its weight deducted from the weight of mixed chlorides. Silica beakers were used for the precipitations involved, and the solution of the alkaline chlorides was evaporated in a large quartz dish. Results were corrected for the sodium chloride found in blank determination on the water and reagents, of which measured amounts were used in the analyses.

19.95 c.c. of solution taken = 0.1805 grm. KCl, 0.1917 grm. NaCl (= 0.3722 grm. NaCl + KCl), 0.005 grm. Al, 0.04 grm. CaCO_3 , 0.04 grm. MgO in hydrochloric solution.

In A, magnesium was removed by barium hydroxide.

In B, magnesium was removed by alcoholic ammonium carbonate (Gooch and Eddy).

In C one grm. of H_2SO_4 added to the solution measured off; precipitated by BaCl_2 at beginning. Magnesium removed by alcoholic ammonium carbonate.

In D, *Direct* determination of potash.

|| In A, the results approach the theoretical quantity; in B, a little higher.

In C, the results are low, but this is unavoidable owing to the inevitable adsorption of alkaline chlorides by the barium sulphate. If the precipitation of sulphuric by benzidine could be applied in such cases, it would be an advantage. Here also the cobaltinitrite process and subsequent treatment of precipitate by perchlorate process may be used (*cf. Bennett loc. cit.*).

In D, the results show that the direct determination of potash gives good results.

	KCl + NaCl found (Grm.)	KCl found (Grm.).	NaCl by Difference (Grm.).	Mean KCl (Grm.).	Mean NaCl (Grm.).
A	0.3716, 0.3731, 0.3725	0.1803, 0.1807, 0.1812	0.1913, 0.1924, 0.1913	0.1807	0.1917
B	0.3738, 0.3726	0.1823, 0.1819	0.1915, 0.1917	0.1821	0.1911
C	0.3637, 0.3648, 0.3690	0.1740, 0.1761, 0.1773	0.1897, 0.1887, 0.1917	0.1758	0.1900
D	—	0.1814, 0.1815	—	0.1814	—

Note.—The results under C confirm the general conclusion that more potash is adsorbed by barium sulphate than soda.

Solubility of Potassium Perchlorate in the Washing Solution.—The following table gives the results of some careful determinations of the solubility of potassium perchlorate in the washing fluid, made as described, and containing 97 per cent. alcohol and 0.2 per cent. HClO_4 :

Temp. C.	Mgrms. per 100 c.c. of Saturated Solution.	Mgrms. per 100 grm. of Solution.	Solubility by Weight in Volume.	Solubility by Weight in Weight.
10°	2.3	2.9	1 : 47,000	1 : 35,000
14°	3.2	3.9	1 : 32,000	1 : 25,000
15°	3.9	4.8	1 : 26,000	1 : 21,000
17.5°	4.1	5.1	1 : 24,000	1 : 19,000
24°	7.3	9.1	1 : 14,000	1 : 11,000
26.5°	11.1	13.6	1 : 9,000	1 : 7,500

The figures by weight in weight have been calculated, taking the sp. gr. of the washing fluid as 0.807.

Most of these results are the average of two closely agreeing duplicate estimations, and the figures are a very fair approximation to the truth when the small amounts of substance actually present are taken into consideration. All results were obtained by evaporating 100 c.c. of the saturated solution, filtered at room temperature, to dryness, transferring the residue by means of a little hot water to a platinum-shaped quartz crucible, evaporating, adding a drop of sulphuric acid, igniting strongly, and weighing as sulphate. This last procedure is necessary owing to the separation

of carbonaceous particles when the alcohol has been evaporated off, so preventing the direct weighing of the residue on evaporation as potassium perchlorate. The weighing of the potassium sulphate was done with the greatest care to 0.0001 gm.

Loss by Solubility of Potassium Perchlorate.—It is obvious that the actual amount lost in washing, under practical conditions, is of more importance than the true solubility of KClO_4 . The following experiments show that, under such practical conditions of washing, the actual loss to be expected is but slight, except when the room temperature rises to 20°C . From the results of Table I. on pure potassium chloride, it is obvious that the mere stirring up of pure potassium perchlorate with the wash liquid and decantation (twice, with 10 c.c. each time) causes no appreciable loss, so that the loss by continued washing of the precipitate on the asbestos is the chief cause of low results.

TWENTY C.C. QUARTZ "GOOCH" USED.

A. Filtered slowly.

Temp. C.	Potassium Per- chlorate in "Gooch" Crucible. (Grm.)	Time of Filtration Per 100 c.c. (Minutes.)	Loss = KClO_4 dissolved out per 100 c.c. (Grm.)
10^{**}	0.64 approx.	6	0.0014
15°	0.64	6	0.0015
15°	0.64	6	0.0017
$19^\circ\dagger$	0.64	6	0.0021
19°	0.64	6	0.0020
21°	0.64	6	0.0022

Average 0.0016 gm. at 15°C .

Average 0.0021 gm. at 19°C .

B. Filtered more rapidly.

15°	0.64	3	0.0012
15°	0.64	3	0.0015
15°	0.64	3	0.0009
21°	0.64	3	0.0016

Average 0.0012 gm. at 15°C .

WITH VARYING AMOUNTS OF POTASSIUM PERCHLORATE (10 C.C. "GOOCH" CRUCIBLE USED.)

15°	0.03	6	0.0005
15°	0.05	6	0.0006
15°	0.09	6	0.0008
15°	0.2	6	0.0012
21°	0.2	6	0.0015

* On passing the filtrate through the crucible again the loss was nil.

† On passing the filtrate through the crucible again the loss was 0.0002 gm.

KClO₄
Dissolved
Per 100 c.c.
(Average.)

Thin and Cumming, using wash liquid containing 97·8 per cent. alcohol
and 0·2 per cent. HClO₄ found 1·8 mgrms.

Thin and Cumming, using wash liquid of pure 98·8 per cent. alcohol
found 4·7 ..

(The amount of KClO₄ in the Gooch was 0·37 grm. and temp. 20° to 22° C.)

Davis, using wash liquid containing 94 per cent. alcohol and 0·2 per cent.

HClO₄ found 2·4 ..

Davis, using wash liquid of pure 95 per cent. alcohol found 14·6 ..

(Weight of precipitate and temperature not given.)

Comparison of Platinum with Perchlorate Process as regards Loss in Washing.—

Davis found that 100 c.c. of 80 per cent. alcohol dissolved out in washing 4·2 mgrms. K₂PtCl₆, equivalent to a loss of 1·3 mgrm. KCl. Now the highest loss of perchlorate I have found per 100 c.c. of washing fluid used = 2·1 mgrms. (at 21° C.), equivalent to a loss of 1·1 mgrm. KCl. (Working at 15° C. the loss [perchlorate process] would be about 0·7 mgrm. KCl.) Evidently the use of the wash liquid described in this paper makes the perchlorate process quite as accurate as the platinum one, and in both cases washing with excessive amounts of liquid will tend to make the results low.

Loss on Washing KClO₄ with a Mixture in Equal Volumes of above Washing Liquid and Pure Ether.—KClO₄ present, 0·64 grm. Time of filtration, six minutes.

Loss per 100 c.c. at 14° C., 0·0008 grm.; at 15·5° C., 0·0008 grm. These figures show a considerable reduction in the solubility when compared with A above.

In this last mixture I find that sodium perchlorate dissolves freely and rapidly.

My thanks are due to Mr. Blount for his helpful interest in this work, and also for his kind permission to give his results as obtained by the platinum process.

These analyses were carried out in the laboratory of Messrs. R. Fry and Co., Ltd., of Brighton.

ADDITIONAL NOTE ON THE OFFICIAL METHOD FOR FERTILISERS, 1918.

(Leaflet 18 of the Board of Agriculture and Fisheries.)

Since 1918 the perchlorate method has been given as an alternative to the platinum process. I here summarise the details and append criticisms.

A preliminary treatment: preparation of the solution for potash estimation.

1. *Potash Salts containing Sulphates.*—The solution is precipitated by barium chloride, and the excess of this removed by the least possible excess of sulphuric acid. An aliquot part of the filtrate is evaporated with hydrochloric, residue dissolved, filtered, and submitted to either the platinum or perchlorate process.

Criticism.—This is a correct treatment if the platinum process is used, but if perchlorate method is used it is better to allow the slight excess of barium chloride to remain, or else traces of the sulphates of sodium, calcium, and magnesium may be

found with the perchlorate weighed. Attention is again drawn to the presence of traces of barium sulphate in the precipitate in such cases.

2. For flue dust: guanos and mixed fertilisers. The preparation of the solution will eliminate nearly everything but sodium and potassium, and may be regarded as satisfactory.

B. Perchlorate Process.—Two evaporations with perchloric acid are prescribed “till white fumes are copiously evolved,” 20 c.c. of 95 per cent. to 96 per cent. alcohol are now added. After stirring and allowing to settle, the liquid is decanted, draining off as completely as possible; a saturated solution of potassium perchlorate in pure alcohol of same strength is used to transfer and to wash precipitate completely. Drying temperature 100° C.

Criticism.—The repeated evaporation with perchloric acid is a good feature. Final evaporation to dryness, or till fumes of perchloric acid have nearly ceased, would be an improvement. A higher percentage alcohol, 97 per cent. at least, should be used. The method I have described is preferable—viz., evaporation to dryness, treatment with 10 c.c. of 97 per cent. alcohol, containing 0.2 per cent. perchloric acid, decantation, warming to drive off alcohol, redissolving in boiling water and again evaporating, a second treatment with 10 c.c. of the same liquid, decanting off, transferring by use of the filtrate to a Gooch, and finally washing with the same perchloric alcohol in small portions (altogether 10 to 15 c.c. for final washings), and drying at 130° C. If, however, suitable apparatus for drawing off the filtrate is not available, then the process may be followed up to the second decantation, and for the rest of washing a saturated solution of potassium perchlorate in alcohol preferably containing 97 per cent. and 0.2 per cent. perchloric acid is used. The precipitate is filtered off, on a Gooch crucible, and dried at 130° C. If filter papers are used, then a neutral solution of potassium perchlorate in alcohol must be employed (to wash out the traces of perchloric remaining), and the drying temperature should be about 120° C.—i.e., as high as the paper will stand without charring. Ten c.c. of 97 per cent. alcohol dissolve about 2 grms. of sodium perchlorate, and alkaline earth perchlorates are also easily soluble. Therefore much washing is unnecessary.

It is important, if the official process is used, that the 20 c.c. of alcohol added should be drained off very completely before the perchlorate solution in pure alcohol is added. Otherwise the trace of HClO_4 remaining will precipitate 2 to 3 mgrms. of KClO_4 from the washing solution. This possibility is avoided if the perchlorate wash solution is made with alcohol containing perchloric acid.

A moderate excess of perchloric acid is sufficient for official process; 10 c.c. of 20 per cent. perchloric will generally suffice for one gram of substance taken.

It is best to use pure duty-paid alcohol. Industrial methylated spirits (strongest) contain an indefinite amount of water, and are, also, too dilute.

Joseph and Martin (*J. Soc. Chem. Ind.*, 1920, 39, 91,) record a violent explosion on concentrating the filtrate from the perchlorate precipitate to low bulk, in order to estimate sodium directly, in the ash of papyrus. There does not seem to be any danger in the determination of potassium, and Schloessing determined the NaCl in mixtures of pure $\text{KCl} + \text{NaCl}$ by evaporation of the filtrate from the

potash, and finally weighing as the sulphate of sodium, apparently without accident.

NOTE.—While Baxter and Kobayashi have shown that at 0° C. the solubility of potassium perchlorate in alcohol containing 0.1 per cent. HClO_4 is practically the same as in alcohol containing 0.2 per cent., still, their own figures for the solubility at 21° C. show that the 0.2 mixture dissolves less than the 0.1 per cent. mixture at room temperatures.



ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Estimation of Free Acetic Acid in Acetylsalicylic Acid. A. N. Smith. (*Pharm. J.*, 1920, 105, 90-94.)—One grm. of the sample is spread in a thin layer on the surface of a piece of fine muslin tied over the top of a funnel 4 inches in diameter; the stem of the funnel is fitted in a hole in a glass plate, and a bell-jar is placed over the whole. A tube leading from the top of the bell-jar extends to the bottom of a vessel containing a layer of water, and this vessel is connected with a water-pump. Air, free from carbon dioxide, is aspirated slowly through the apparatus for one hour, and the acetic acid absorbed in the water is then titrated. Salicylic acid does not volatilise under these conditions. It is suggested that the maximum permissible limit for free acetic acid in acetylsalicylic acid should be 0.05 per cent., and for free salicylic acid 0.1 per cent., and double these amounts for tablets of the substance.

W. P. S.

Assay of Aconite Preparations. E. J. Chappel and N. L. Allport. (*Pharm. J.*, 1920, 105, 94.)—Fifteen c.c. of the liniment or 100 c.c. of the tincture are evaporated at a low temperature to remove most of the alcohol, 5 c.c. of 10 per cent. sulphuric acid and 20 c.c. of water are then added, the mixture transferred to a separating funnel and extracted with two successive quantities of 20 c.c. of petroleum spirit (b.p. 40° to 60° C.). The united petroleum spirit extracts are washed twice with water; this water is added to the acid solution, which is then rendered ammoniacal and extracted four times with ether. The ethereal extracts are washed once with water, evaporated to dryness, the alkaloidal residue is dried at a low temperature to ensure removal of all ammonia, and then titrated in the usual way.

W. P. S.

Estimation of Allyl Mustard Oil in Black Mustard. D. Raquet. (*J. Pharm. Chim.*, 1920, 22, 92-93.)—By digesting mustard flour with dilute alcohol at 30° to 35° C. for an hour the same result is obtained as after six hours' extraction with cold alcohol, but maceration with water, as prescribed by the French Codex,

gives inaccurate results. By heating the mixture of ammoniacal silver nitrate and mustard oil for an hour on the water-bath the whole of the sulphur will be converted into silver sulphide. These modifications of the Codex method will increase the yield of mustard oil by 20 to 38 per cent. If they are adopted, it will be necessary to raise the minimum standard for mustard oil in mustard from 0.7 to 0.9 per cent.

C. A. M.

Detection and Estimation of Neutralising Agents in Butter and Similar Products. L. W. Ferris. (*J. Ind. and Eng. Chem.*, 1920, 12, 757-760.)—Since it is not practicable to remove all the milk salts from butter, the salts in butter from neutralised cream will differ from those in butter from normal cream. On treating the sample with a known quantity of $\frac{N}{10}$ hydrochloric acid and an excess of picric acid (to precipitate proteins), and extracting the lactic acid and excess of picric acid with ether, a solution of the salts will be left. When this is titrated with sodium hydroxide solution, with methyl orange as indicator, the excess of hydrochloric acid added over the amount neutralised by the sodium hydroxide will be the alkalinity of the salts, or the amount which has combined with the bases formerly present as alkalis, lactates, and phosphates. In normal milk and milk products the alkalinity of the salts present shows a definite ratio to the inorganic phosphoric acid, being approximately 45 c.c. of $\frac{N}{10}$ -alkali solution per 1 grm. of phosphoric anhydride. In neutralised milk this ratio increases with the amount of alkali which has been used. Butter made from neutralised cream will show an alkalinity ratio similar to that of the cream from which it was made, provided that the cream, when churned, contains sufficient lactic acid to keep the phosphate in solution, otherwise the alkalinity ratio may be lower than that of the cream. About one pound of the butter is melted at 45° C., and rendered homogeneous, and the water estimated. One hundred grms. are shaken in a stoppered bottle with warm petroleum spirit until the fat has dissolved. The bottle is centrifuged, the fat solution siphoned off, and the residue shaken with 50 c.c. of $\frac{N}{10}$ hydrochloric acid and 100 c.c. of a saturated solution of picric acid continually for thirty minutes, or, at intervals, for two hours. It is then cooled and filtered, and 50 c.c. of the filtrate extracted for twenty hours with ether, after which it is transferred to an Erlenmeyer flask into which fits a tube with a double bulb with a constriction between so that the condensed vapours form a seal above the flask, while the steam is washed by the returning condensed liquid. After being gently boiled to expel all carbon dioxide the liquid is cooled and titrated with $\frac{N}{10}$ sodium hydroxide solution, with methyl orange or butter yellow as indicator. In calculating the amount of $\frac{N}{10}$ hydrochloric acid in the 50 c.c. of filtrate, the total volume of the solution is taken as 150 c.c. plus the water in the sample. The result less the $\frac{N}{10}$ alkali used in the titration gives the alkalinity of the 50 c.c. The solution is then treated with 10 c.c. of magnesia mixture and 10 c.c. of strong ammonia solution, allowed to stand over-night, and filtered. The precipitate is washed twice with 2.5 per cent. ammonia solution, and dissolved in hot dilute nitric acid, and the phosphoric anhydride is estimated volumetrically. The alkalinity as c.c. of $\frac{N}{10}$ alkali divided by the mgrms. of P_2O_5 in the 50 c.c., and multiplied by 100, gives the "alkalinity ratio." In the case of milk, butter-milk, or thin cream, 15 grms. are

taken; for thick cream, 25 grms.; for evaporated milk, 7 grms.; and for dry milk, 1 grm. Samples of neutralised cream and butter prepared from it showed alkalinity ratios ranging from 81.1 to 221.8. An approximate estimation of the acidity neutralised in butter and allied products may be made by means of the formula—

$$A = \frac{P(R-45)}{11.11},$$

where A represents the percentage of lactic acid neutralised, P the percentage of P_2O_5 in cream or milk, and R the ratio of the sample. When this formula is applied to butter, and the amount of phosphoric anhydride in the cream from which it was made is not known, an average figure must be taken. C. A. M.

Kreis Reaction of Cottonseed Oil Products. W. B. Smith. (*J. Ind. and Eng. Chem.*, 1920, **12**, 764-766.)—The Kreis test for rancidity, in which the oil or fat is shaken with equal volumes of hydrochloric acid and phlogluinol-ether solution, gives no coloration with fresh olive, sesame, coconut, palm, and neatsfoot oils, or with tallow or lard. Crude cottonseed oil, however, contains a chromogenic substance which gives a coloration with the reagent more pronounced than any coloration due to rancidity in other oils. Contrary to the experience of Kerr (*ANALYST*, 1918, **43**, 327), refining the oils does not always remove the reacting substance, which may also be found in certain cottonseed products. Hence the Kreis reaction is not absolutely trustworthy for the detection of rancidity in cottonseed oil. In some cases exposure to sunlight destroys the cottonseed chromogenic substance, so that oils which give a pronounced reaction before exposure may give a negative one afterwards. In this way it is sometimes possible to distinguish between rancid and non-rancid oils giving a positive Kreis reaction. C. A. M.

Method for the Estimation of Cineol in Eucalyptus Oils. T. T. Cocking. (*Pharm. J.*, 1920, **105**, 81-83.)—Three grms. of the eucalyptus oil, dried previously over calcium chloride, are mixed with 2.1 grms. of *o*-cresol, and the solidifying point of the mixture is determined. Reference to a graph constructed from the results of estimations of mixtures containing known amounts of cineol gives the quantity of cineol present in the oil under examination. Terebene may be used as the diluent for the cineol in making the standard mixtures. W. P. S.

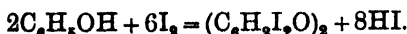
Detection of Inorganic Phosphate in Glycerophosphates. J. L. Lizius. (*Pharm. J.*, 1920, **105**, 100-101.)—When 0.8 grm. of the glycerophosphate is mixed with 10 c.c. of dilute nitric acid and 10 c.c. of 10 per cent. ammonium molybdate solution, the presence of 0.1 per cent. or more of inorganic phosphate is indicated by the formation of a turbidity. Attention is directed to the solubility of the yellow ammonium phosphomolybdate in excess of either phosphate or glycerophosphate, and the unreliability of the test prescribed in the U.S. Pharmacopœia is possibly due to this fact. A sensitive reagent for the purpose has been described by Scott and Plimmer (*J. Physiol.*, 1906, **35**, 120); it is prepared by adding 80 c.c. of 10 per cent. ammonium molybdate solution to 12 c.c. of hydrochloric acid (sp. gr. 1.16), dissolving 20 grms. of ammonium chloride in the mixture, and then adding 10 c.c. of saturated

potassium persulphate solution. Five c.c. of this reagent are mixed with 0.1 gm. of the substance to be tested; a precipitate forms within a few minutes if the substance contains as little as 0.001 per cent. of inorganic phosphate. W. P. S.

Estimation of Ipecacuanhic Acid in Ipecacuanha Preparations. R. Huerre. (*J. Pharm. Chim.*, 1920, 21, 425-433.)—The method depends on the precipitation of ipecacuanhic acid by saturated salt solutions, ammonium sulphate giving the best results. A definite weight of ipecacuanha extract is dissolved in water, the solution treated with ammonium sulphate in quantity slightly more than is required for saturation, the precipitate is collected, and washed with saturated ammonium sulphate solution; the still moist precipitate is then dissolved on the filter in 90 per cent. alcohol, the alcoholic solution is evaporated to dryness, the residue dissolved in water, the solution filtered, and the filtrate diluted to a definite volume. An aliquot portion of this solution is evaporated to dryness, the residue of ipecacuanhic acid heated at 100° C., and weighed. An allowance must be made for the small quantity of ammonium sulphate still present; for this purpose another aliquot portion of the solution is acidified with hydrochloric acid, and the sulphate is precipitated as barium sulphate. Powdered ipecacuanha contains from 3 to 4 per cent. of ipecacuanhic acid. W. P. S.

Assay of Opium. A. Jermstadt. (*Inaugural Dissertation, Basle*, 1920; through *Pharm. J.*, 1920, 105, 172.)—Critical examination of official and unofficial methods for the estimation of morphine in opium showed that the lime method adopted in the British, American, French, Dutch, and other Pharmacopœias does not always give concordant results; the morphine separated is impure, but when it is freed from narcotine and calcium meconate by treatment with benzene and alcohol the results obtained are more trustworthy. The unofficial methods were found to be unsatisfactory. The following method is recommended: 6 grms. of powdered opium are mixed to a paste with 6 grms. of water, the paste is rinsed into a weighed flask, and water is added to make the contents weigh 54 grms. The mixture is shaken frequently during thirty minutes, then filtered, 40 grms. of the filtrate are mixed with 2 grms. of $\frac{N}{1}$ ammonia solution, and the mixture at once filtered. Thirty-six grms. of this filtrate are shaken with 7 grms. of ether and 4 grms. of $\frac{N}{1}$ ammonia solution for ten minutes, 10 c.c. of ether are then added, and after thirty minutes the ethereal layer is decanted through a filter; the extraction is repeated with a further 10 c.c. of ether. The contents of the flask are then poured on to the filter, the flask and filter are washed with three successive portions of 5 c.c. of water saturated with ether, and the flask and filter are dried at 100° C. The contents of the filter are then transferred to the flask, dissolved in 25 c.c. of $\frac{N}{10}$ hydrochloric acid, the solution is poured through the filter, the flask and filter are washed with about 75 c.c. of water, and the excess of acid is titrated with $\frac{N}{10}$ sodium hydroxide solution, using methyl red as indicator. Each c.c. of $\frac{N}{10}$ hydrochloric acid is equivalent to 0.0285 gm. of anhydrous morphine. W. P. S.

Estimation of Phenol and Salicylic Acid in B.P. Ointments. G. D. Elsdon. (*Pharm. J.*, 1920, 105, 86.)—The method is an application of the iodine reaction described by Wilkie (*ANALYST*, 1911, 36, 295) :



From 0.5 to 0.6 gram. of the phenol ointment is dissolved in 5 c.c. of warm chloroform, 25 c.c. of $\frac{N}{10}$ sodium carbonate are then added, and the mixture is boiled and shaken. The shaking is continued while 25 c.c. of cold water are added; the mixture is then cooled, 25 c.c. of $\frac{N}{10}$ iodine are added, and the mixture again shaken. After five minutes the mixture is acidified slightly with sulphuric acid, and the excess of iodine is titrated with $\frac{N}{10}$ thiosulphate solution. Each c.c. of $\frac{N}{10}$ iodine solution is equivalent to 0.001568 gram. of phenol. The same procedure is adopted with salicylic acid ointments; 1 c.c. of $\frac{N}{10}$ iodine solution is equivalent to 0.002301 gram. of salicylic acid.

W. P. S.

Estimation of Phenolphthalein as Tetraiodophenolphthalein. S. Palkin. (*J. Ind. and Eng. Chem.*, 1920, 12, 766-769.)—Errors in the estimation of phenolphthalein as tetraiodophenolphthalein, due to oxidation, may be eliminated by keeping the temperature low. The alkalinity is the most important factor in the completeness of the reaction, but to obtain quantitative results it is necessary to redissolve the precipitate by means of potassium hydroxide and again precipitate it with acid, and to repeat this process several times. The phenolphthalein (not exceeding 0.25 gram.) is dissolved in the minimum quantity of potassium hydroxide solution (about 30 per cent.), and the solution chilled by the addition of 15 to 20 grms. of ice, and treated with excess of 10 per cent. iodine reagent (iodine, 10 per cent., dissolved in 15 per cent. potassium iodide solution, and the free iodine discharged with alkali). The precipitate is dissolved by potassium hydroxide solution, added drop by drop from a burette, then reprecipitated with strong acid, and this process is repeated alternately three or four times. Finally, the alkaline solution is transferred to a separating funnel, chilled with ice, treated with 0.5 c.c. of 15 per cent. sodium sulphate solution, and acidified with strong hydrochloric acid. The precipitate is extracted with 50 to 75 c.c. of a mixture of acetone and chloroform (1 : 3), the extract drawn off, and the shaking twice repeated with 35 c.c. of the mixed solvents. The extracts are washed with water in a second funnel, transferred to a tared Erlenmeyer flask, and evaporated to dryness, and the residue is dried for twenty minutes at 100° C. and weighed. The weight multiplied by 0.3871 gives the amount of phenolphthalein. Medicinal preparations of phenolphthalein which do not contain any substances forming compounds soluble in chloroform acetone may be analysed directly by this method. But in the case of preparations containing chocolate or other constituents soluble in the mixed solvents preliminary treatment is necessary. Extraction of such tablets with petroleum spirit for about two hours will remove the fat, and the residue should then be extracted for at least an hour with pure acetone, dried at 100° C., and dissolved in alkali solution, the liquid made up to 50 c.c. and filtered, and an aliquot portion of the filtrate is taken for the estimation of the phenolphthalein as described.

C. A. M.

Estimation of Starch enclosed in Cellular Tissue by Means of Ammoniacal Copper Oxide Solution. F. Kaulfersch. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1920, 39, 344-346.)—During the analysis of an anchovy paste containing starch derived from ground beans it was found that a considerable proportion of the starch was enclosed in cellular tissue, and resisted the action of diastase and hydrochloric acid. The following method was, therefore, used for the estimation: 0.5 gm. of the dry, fat-free paste was stirred for some minutes with ammoniacal copper oxide solution, the copper then removed by treatment with hydrogen sulphide, and the solution boiled to expel excess of the latter. The solution was then heated at 65° C. for six hours with the addition of diastase, filtered, boiled under a reflux condenser for three hours with the requisite quantity of hydrochloric acid, neutralised, and the copper-reducing power determined. In this way the starch content was found to be 36.4 per cent.; when the treatment with the copper solution was omitted the result was 24.3 per cent.

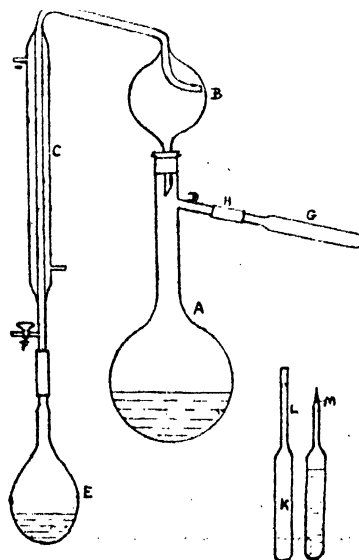
W. P. S.

Balance of the Specific Gravity of Wine. W. J. Baragiola and O. Schuppli. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1920, 39, 313-335.)—The sp. gr. of the aqueous solution of the non-alcoholic constituents of wine, as calculated from the sp. gr. of some twenty-five separate constituents, was found to be 1.0072, whilst direct determination showed the sp. gr. to be 1.0085. This difference of 0.0013 would correspond with the presence of about 3 grms. per litre of unknown or non-estimated constituents. The fact that the direct and indirect methods of estimating total solids in wine yield concordant results is probably due to the fact that the tables used in the indirect method have been compiled from the results of actual estimations.

W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Estimation of Carbon Dioxide and Fermentable Sugars. A. Slator. (*J. Soc. Chem. Ind.*, 1920, 39, 149-151.)—The apparatus described was devised originally to estimate the total amount of carbon dioxide formed when sugars are fermented by yeast. It was subsequently found to be of general use, and can be employed in most cases where it is necessary to estimate this gas. The apparatus consists of a distillation flask, A, connected to a condenser, C, in the manner shown in the diagram. The large trap, B, prevents spray and froth being carried over during the distillation of carbon dioxide from A to the "assay" flask, E, which contains standard baryta water. G is a test-tube to which has been sealed a piece of glass tubing. This tube can be connected to the side piece, D, by means of pressure tubing, H. The whole apparatus can be exhausted through the stopcock, F. The estimation of carbon



dioxide in sodium carbonate is carried out in the following manner: 25 c.c. of $\frac{N}{10}$ sodium carbonate are placed in *A*, and *G* is filled with dilute sulphuric acid. Into *E* are introduced 30 c.c. of $\frac{N}{10}$ baryta water. The apparatus is exhausted, a screw clip being placed at *H* to prevent the acid in *G* passing into *A*. *F* is then closed and the clip removed. The acid in *G* is warmed and boils into *A*. Both *A* and *G* are then well boiled and *E* shaken to absorb the gas. When the distillation is complete, air free from carbon dioxide is allowed to enter the apparatus through *F*. The excess of baryta in the turbid solution in *E* is then titrated with standard hydrochloric acid, using phenolphthalein as indicator. The total carbonate in drinking-water can be determined by placing dilute sulphuric acid in *A*, while *G* is replaced by a funnel through which the water to be analysed is run into the exhausted apparatus; 250 c.c. of a certain water yielded CO_2 , equivalent to 14.25 c.c. of $\frac{N}{10}$ alkali. The same water showed 15.5° temporary hardness (parts CaCO_3 per 100,000) by direct titration, using methyl orange as indicator. Carbon dioxide in calcium carbonate may be estimated by distillation with phosphoric acid, while free CO_2 in liquids is determined by filling *G* with the liquid and then distilling off the CO_2 . Carbon dioxide formed in certain oxidation reactions may also be collected and estimated by means of this apparatus, an example being potassium tetroxalate. $\text{KH}_2(\text{C}_2\text{O}_4)_2 \cdot 2\text{H}_2\text{O}$ may be oxidised in the apparatus with slight excess of acidified permanganate solution. Special precautions must be observed if volatile acids other than carbonic are present, and if insoluble salts are present care must be taken to insure all the carbonate being acted upon by the acid. The estimation of sugars by alcoholic fermentation affords another opportunity for using the apparatus. Into tubes (*K*) were introduced measured quantities of solutions of pure sugars, 2 c.c. of yeast water (sp. gr. 1.017), and water to make up the volume to about 12 c.c. The tubes were plugged with cotton-wool and then sterilised. Each tube was seeded with a pure culture of yeast, the tube heated at *L*, and the glass pulled out to a fine capillary. The tube was then exhausted and sealed at *L*. Fermentation was allowed to proceed at 25°C ., the tubes being shaken each day. The carbon dioxide produced was estimated in the following way: A file mark was made at *M* on the tube, which was then connected to the apparatus. After exhausting the flask and boiling the water in *A*, *F* was shut off and the end of the tube broken at *M* in the pressure tubing. The tube and flask were then both warmed and all the carbon dioxide collected in *E*. Claim, of course, is not made that the sugar is completely fermented according to the equation $\text{C}_6\text{H}_{12}\text{O}_6 = 2\text{CO}_2 + 2\text{C}_2\text{H}_5\text{OH}$. Probably about 95 per cent. of the CO_2 is produced according to this equation; another 2 per cent. comes from the auto-fermentation of the yeast, and the rest from the auto-digestion of the yeast. The apparatus was also applied in an attempt to estimate some of the constituents of ordinary brewery wort. The total amount of fermentable carbohydrates is determined by fermenting wort with yeast, filtering, and then distilling off the alcohol from a measured volume. The residue is made up again to this volume and the process repeated. From the gravity of the final residue the total amount fermented may be calculated. In a fermentation of this kind about 95 per cent. of the theoretical yield of alcohol is obtained, and in general it is found that the total amount of fermentable carbohydrates may be obtained in this manner with a fair

degree of accuracy, but it is advisable to carry out control experiments with pure sugars before using the method for any special purpose. The presence of certain enzymes in yeast serves to characterise and identify different races. The following method is recommended for carrying out such tests: For the detection of invertase, tubes containing yeast water, cane sugar (0.2 grm.), and dextrose (0.02 grm.) are inoculated with a pure culture of the yeast. The tubes are exhausted and sealed up. After about a week the carbon dioxide is distilled off and estimated. If no invertase is present in the yeast, carbon dioxide to neutralise about 4 c.c. of alkali is obtained; if present, up to 40 c.c. to 50 c.c. is neutralised. The presence of dextrose is an advantage for growth, and a certain amount of fermentation is insured. Maltose can be detected by using maltose in place of cane sugar. The presence of or absence of galactozymase can be shown by using galactose. The following results were obtained:

Yeast.	Enzyme.	Alkali Neutralised.	Presence or Absence of Enzyme.
Apiculatus	Invertase	c.c. 3	-
"	Maltase	3.7	-
Cerevisiæ	Galactozymase	31	+
Wild yeast, A	"	34	+
" " B	"	2.5	-
" " C	"	32	+

H. F. E. H.

Estimation of Volatile Acids in Fermentation Products. G. Hinard (*Ann. Chim. anal.*, 1920, 2, 239-242.)—Fifty c.c. of the sample, together with 10 c.c. of water, are distilled from a flask fitted with a bulb tube connecting with a condenser; 50 c.c. of distillate are collected and titrated. Fifty c.c. of water are then added to the distillation flask, and a further 50 c.c. distilled and neutralised; these operations are repeated several times. When a curve is constructed from the results, the volumes distilled being the abscissæ and the successive quantities of alkali solution used for the neutralisations the ordinates, the asymptote of the curve indicates the volume of alkali solution necessary for the neutralisation of the total volatile acids present.

W. P. S.

Colorimetric Estimation of Glycogen. R. Thieulin. (*J. Pharm. Chim.*, 1920, 21, 91-93.)—In aqueous solution glycogen gives a red-brown coloration with iodine, the intensity of the colour being proportional to the amount of glycogen, provided a small definite amount of iodine is used. To estimate glycogen in liver, the latter is treated with 60 per cent. potassium hydroxide solution, the glycogen then precipitated by the addition of an equal volume of alcohol, the precipitate is separated by centrifugal action, washed with alcohol, and dissolved in a small quantity of water (for 3 grms. of liver 10 c.c. of water will be sufficient). The

solution is neutralised with acetic acid, and 2 c.c. of it are transferred to a test-tube and treated with two drops of iodine solution (iodine, 1 grm.; potassium iodide 2 grms.; water, 20 c.c.). The coloration obtained is compared with those given by definite amounts of glycogen, each dissolved in 2 c.c. of water and treated with two drops of the iodine solution.

W. P. S.

Acidity of Ropy Milk. K. Freear and E. C. V. Venn. (*Biochem. J.*, 1920, 14, 422-431.)—The paper describes the result of work done with the organisms found in samples of ropy milk from two Lancashire farms. They were plated out upon neutral "Lemco" agar, and from both of them a Gram-positive coccus showing also diplococcal forms, and in fluid cultures short chains, was obtained. These appeared to be identical with, or closely related to, the *Streptococcus Hollandicus*, and for comparative purposes strains of this organism were also employed. The experiments show that these strains produced ropiness in milk, associated with a minimum acidity, as determined by $\frac{N}{10}$ NaOH of 0.43 per cent. lactic acid, and that the ropiness continued for varying periods of time, accompanied by an acidity which never rose above 1.0 per cent. (except on one occasion). In every case the ropiness eventually diminished, and when studied for sufficiently long periods entirely disappeared, but these changes were not accompanied by any material alteration in the acidity. The results obtained by the NaOH titration method were confirmed by electrometric determinations, from which it appeared that ropiness was found in milk with a P_H range of 5.82 to 4.10. With diminishing ropiness no appreciable alteration in the P_H value could be detected, although in one case the experiment was continued for seventeen days, by which time ropiness had disappeared.

H. F. E. H.

Mutton Bird Oil. N. Evers and H. J. Foster. (*Pharm. J.*, 1920, 105, 100.)—Two samples of oil received from Tasmania and New Zealand respectively, and stated to be mutton bird oils (*Aestrelata lessona*), had the following physical and chemical characters:

	Sample from Tasmania.	Sample from New Zealand.
Appearance...	Clear brown liquid	Yellowish-brown semi-solid
Odour and taste ...	Slightly fishy	Fishy
Sp. gr. at 15.5°/15.5° C. ...	0.8835	0.9179 (10°/15.5° C.)
Iodine value ...	132	99.5
Saponification value ...	125.9	200
Acid value ...	5.7	6.8
Unsaponifiable matter ...	31.1 per cent.	0.98 per cent.
Ether-insoluble bromides ...	10.4 per cent.	None

The figures for the New Zealand sample raise doubts as to whether it is mutton bird oil; those for the Tasmanian sample, with the exception of the iodine value, agree with the results recorded previously by L. H. Smith (*ANALYST*, 1911, 36, 290). In any case, the oils bear little resemblance to cod-liver oil, for which they have been suggested as substitutes.

W. P. S.

Determination of Grade of Peat. G. Keppeler. (*Mitt. Ver. Förder. Moorkultur in Deuts. Reiche*, 1920 [1]; through *Chem. Zentr.*, 1920, **91**, II., 548.)—The degree of conversion to peat of mosses can be determined by decomposing the material with 72 per cent. sulphuric acid. The polysaccharides pass into solution, and the sugars can be estimated by titration with Fehling's solution. The dextrose estimated in this way, and calculated on the water- and ash-free peat, is termed the "total reduction." The maximum possible value is 68 per cent., so that if the "total reduction" for any peat be g , then $100 - g \times 100 \div 68$ is a measure of the degree of conversion of the original vegetable matter into peat. This value is called the "degree of decomposition." The degree of conversion to peat may also be estimated from the residue after treatment with 72 per cent. sulphuric acid. From this residue (referred to 100 parts of organic matter) the ash, and also the residuum left in the acid treatment of sphagnum moss (11 per cent.), are deducted in order to obtain the "degree of conversion." The newer moss peats generally show a low "degree of conversion" (scarcely 30 per cent.), whilst the older ones give a much higher value (up to 50 per cent.).

Pectins in Various Plants. A. J. W. Hornby. (*J. Soc. Chem. Ind.*, 1920, **39**, 246r.)—Examination of the substances not usually determined in the analysis of cattle foods showed that the methyl pectate content of different parts of fresh plants varied greatly when estimated by Fellenberg's method (*ANALYST*, 1918, **43**, 37). As the methyl pectate is partly decomposed on drying, the author has determined the percentage of this substance in a number of fresh food materials and in different parts of the same vegetable. It was found that a much larger proportion exists in the epidermal tissue of the roots, etc., than in the cortex, and the proportion is increased in the parts attacked by insects or otherwise mechanically injured, and also in the portions of the potato tuber exposed to light. It is suggested that this pectin has a protective effect against insect attack owing to the ease with which methyl alcohol is split off from the molecule, this alcohol acting as a strong irritant to insects. The results obtained may indicate that a methyl compound would form an efficient spray for the prevention of insect attacks, but at present the cost would be prohibitive. Tables are given showing numerous results obtained by the above method.

T. J. W.

Estimation of Urea by Means of Xanthydroxyl. Frenkel. (*Ann. Chim. anal.*, 1920, **2**, 234-239.)—For the estimation of urea in urine 10 c.c. of a 10 per cent. solution of the latter are acidified with 35 c.c. of glacial acetic acid, and 1 c.c. of 10 per cent. xanthydroxyl solution (in methyl alcohol) is added while the mixture is stirred; after ten minutes a further 1 c.c. of the reagent is added, and so on until 5 c.c. in all have been added. The precipitate formed is collected after one hour, washed with about 20 c.c. of alcohol, dried at 100° C., and weighed. The weight divided by seven gives the amount of urea. Other substances present in urine do not interfere. The method may be applied to the estimation of urea in blood, after the blood has been treated with a reagent, consisting of mercuric chloride 2.71 grms., potassium iodide 7.20 grms., glacial acetic acid 66 c.c., and water 100 c.c. The

large excess of xanthydrol required for the precipitation of urea may be recovered from the filtrate from the urea precipitate by heating the filtrate until the alcohol has been expelled, neutralising the residual solution with sodium carbonate, and adding chloroform which dissolves the xanthydrol. The chloroform solution is evaporated, and the residue of xanthydrol dissolved in methyl alcohol for further use.

W. P. S.

Nature of Yeast Fat. I. S. MacLean and E. M. Thomas. (*Biochem. J.*, 1920, 14, 483-493.)—The fat examined was obtained from the following sources: (1) Bakers' yeast supplied from the manufacturers, (2) pure culture of brewery yeast, and (3) specimens of brewery yeast. After describing the method of extraction for the fat, the authors deal with its composition. The determination of its iodine value shows that the figure varies from 121 to 175, while the saponification values show wide variation ranging from 151 to 199, depending upon the proportion of sterol present. The constituents of the yeast fat are then discussed, including an account of the fatty acids, the preparation of the methyl esters, identification of the saturated and unsaturated acids, together with an account of the preparation of sterol and its iodine value. The following conclusions were reached: (1) Palmitic, oleic, and linoleic acids have been identified with certainty in yeast fat. (2) The pentadecolic acid, previously described, consists of a mixture of palmitic and lauric acids. Evidence of the presence of aluric acid has been obtained. (3) The presence of an acid melting at 77° C. has been confirmed, the m.-pt. of which agrees with that of arachidic acid. (4) No confirmation of the presence of the dodecenic acid described by Hinsberg and Roos was obtained, and the possibility of this being a mixture of lauric with oleic and linoleic acids has not yet been excluded. (5) A sterol is present partly in the free state and partly as fatty acid esters; this may constitute 20 per cent. of the total yeast fat. (6) The sterol present in yeast appears to be identical with the ergosterol isolated from ergot by Tanret. (7) The variation in m.-pt. described by different observers seems to be due to differences in the purity of the substance, but no conclusive evidence of the presence of a second sterol in yeast has yet been obtained. (8) It seems probable that the mycosterol isolated by Ikeguchi from certain fungi is also identical with ergosterol, and that ergosterol is characteristic of the whole group of cryptogams, just as cholesterol is of the animal, and phytosterol of the higher plant kingdom. (9) Yeast sterol is differentiated from the sterols of the higher plants and animals by the presence of three double bonds in its molecule.

H. F. E. H.

Zinc in the Human Organism. S. Giaya. (*J. Pharm. Chim.*, 1920, 22, 85-92.)—The results of about fifty estimations of zinc in various organs and secretions of normal human subjects are described. The organic matter was destroyed by treatment with potassium chlorate and hydrochloric acid or by means of nitric and sulphuric acid. The liquid was treated with hydrogen sulphide and filtered, the filtrate concentrated to remove excess of hydrochloric acid, diluted, and treated with ammonia in slight excess. It was then acidified with acetic acid, treated with a few c.c. of 10 per cent. sodium acetate solution, again

saturated with hydrogen sulphide, and allowed to stand for forty-eight hours. The zinc sulphide (containing a little iron) was then separated, washed with 5 per cent. acetic acid, saturated with hydrogen sulphide, and dissolved in dilute hydrochloric acid. The solution was evaporated nearly to dryness, the residue boiled with dilute hydrochloric acid and a slight excess of bromine, and the iron precipitated with ammonia. The zinc was then reprecipitated as sulphide as before, the zinc sulphide separated after forty-eight hours, washed, and dissolved in dilute hydrochloric acid, this solution again evaporated to dryness, and the residue dissolved in very dilute acetic acid. The zinc in this solution was finally estimated by titration with potassium ferrocyanide. The amounts of zinc thus found in the total viscera varied from 10 to 50 mgrms., whilst urine contained only traces (0.00017 gm. per litre). The amounts increased with the age of the subject, the viscera of an infant of three months containing only 0.009 gm. These quantities, even when they reach 0.005 gm. per 100 grms. of material, are not sufficient to interfere with toxicological examinations.

C. A. M.

ORGANIC ANALYSIS.

Colorimetric Estimation of Adrenalin. W. L. Scoville. (*J. Ind. and Eng. Chem.*, 1920, 12, 769-771.)—To 20 c.c. of water in a small flask are added 5 c.c. of 1 per cent. potassium iodate and 0.25 c.c. of $\frac{N}{4}$ hydrochloric acid. After warming to 38° C., 0.5 c.c. of standard adrenalin solution is added, and the mixture heated for fifteen minutes. To a similar mixture is added 0.5 c.c. of the (1 : 1000) solution to be tested (or an equivalent volume of a weaker solution), which is then warmed as above. After cooling, the colours of the two solutions are compared in a colorimeter. Should the colour intensities vary more than 25 per cent. it is advisable to repeat the test, using more or less of the unknown adrenalin solution.

The standard adrenalin solution should be freshly prepared by dissolving 0.05 gm. pure adrenalin in 0.5 c.c. $\frac{N}{4}$ hydrochloric acid and diluting to 50 c.c. If the solution to be compared contains bisulphite, 0.05 gm. of pure sodium bisulphite should be added to the standard solution.

A standard colour solution may also be prepared by mixing 10 c.c. of 2.5 per cent. crystallised cobalt chloride solution with 80 c.c. of water containing 2 grms. ammonium carbonate and diluting to 100 c.c. The colour of this standard closely matches that of the adrenalin solution in depths ranging from 25 to 30 mm., but with solutions containing bisulphite the results obtained are too low.

For the estimation of adrenalin in dried glands, 0.1 gm. of the gland is digested at 38° C. for half an hour in 20 c.c. of water, to which has been added 5 c.c. of the iodate solution and 0.25 c.c. of $\frac{N}{4}$ hydrochloric acid. After filtration the solution is transferred to the colorimeter and compared with the standard. From the result obtained the quantity of gland required to contain approximately 0.5 mgrm. of adrenalin may be calculated, and the estimation repeated, using this amount. The results compare very favourably with those obtained by the biological test.

T. J. W.

Rapid Volumetric Methods for the Estimation of Amino-Acids, Organic Acids, and Organic Bases. F. W. Foreman. (*Biochem. J.*, 1920, 14, 451-473.)

—*Preparation of Solution*: A known weight of the amino-acid, or its suitable salt, or of an amino-acid mixture, is dissolved in water free from CO_2 , and made up to a known volume, so that the resulting solution is approximately $\frac{N}{10}$ in strength in relation to the carboxyl-groups. In cases such as tyrosine and cystine, where the amino-acid is insoluble in water, or soluble in too large a volume of water, $\frac{N}{10}$ HCl or $\frac{N}{10}$ soda or other appropriate means must be used for bringing them into solution, so that no separation occurs on adding alcohol. When only a very small quantity of the amino-acid is available it may be weighed accurately into a suitable vessel, dissolved in the desired amount of water, and the whole of the solution used. *Stage 1, Titration in Water*: A 5 c.c. or 10 c.c. portion of the solution is titrated with aq. $\frac{N}{10}$ soda to phenolphthalein (soda standardised to phenolphthalein). *Stage 2, Titration in Alcohol*: Five c.c. or 10 c.c. of the original solution are transferred to a conical flask of about 250 c.c. capacity, ten volumes of 97 per cent. alcohol and three drops of phenolphthalein solution added, and the mixture titrated in daylight over a white plate with $\frac{N}{10}$ alcoholic potash (standardised to phenolphthalein) until a light pink colour is produced. The endpoint is usually obtained quite sharply on adding the last one or two drops of standard alkali (two drops = 0.05 c.c.). A correction is made for the original acidity of the alcohol. *Stage 3, Titration in Alcoholic Formaldehyde*: After obtaining the reading in Stage 2, the same liquid is used for Stage 3. Twelve and a half c.c. of aqueous formaldehyde solution prepared by diluting colourless formalin with two volumes of distilled water, and neutralising the mixture to phenolphthalein, are added for each 50 c.c. of alcohol used in Stage 2, and the titration continued to the same endpoint as before. A mixture of neutralised diluted formalin and alcohol in the same proportions as they have been used is then titrated, and the result obtained on completing Stage 3 is corrected accordingly. The acidity of rectified spirit is very slightly increased on adding the neutral formaldehyde solution. The result obtained in Stage 1 gives useful information when dealing with the dibasic amino-acids, arginine, and salts of amino-acids. The Stage 2 titration value includes the reading obtained in Stage 1. Several amino-acids are correctly estimated in Stage 2. The increase in titration value obtained in Stage 3 gives an idea of the character of the amino-acid, and, when dealing with an amino-acid mixture, affords an indication of the amount of dibasic amino-acids and proline present. The carboxyl-groups of all the amino-acids contained in an amino-acid mixture, except that of arginine, are estimated by the total titration value obtained on completing Stage 3. Ammonia, primary, secondary, and tertiary amines, and basic methylene derivatives of secondary amines do not form ionisable compounds with phenolphthalein in alcoholic solutions containing water, if the concentration of alcohol is sufficiently high. In aqueous alcoholic solutions of the salts of these bases the acid radicles can be titrated accurately with $\frac{N}{10}$ alkali, using phenolphthalein as indicator, if more than about 80 per cent. alcohol is present. When aqueous alcoholic solutions of certain amino-acids containing about 85 per cent. alcohol are titrated with standard alcoholic potash, the amino- or imino-groups liberated from their "internal salt" combinations resemble ammonia and the amines in showing no basicity to phenolphthalein, and the

carboxyl-groups are accurately estimated. Other amino-acids, more particularly dibasic amino-acids and proline, give low results when titrated in alcohol under these conditions, possibly owing to loose combination of alcohol with a carboxyl-group, or loose condensation. The subsequent addition of formaldehyde or acetone, however, results in a disturbance of the equilibrium, so that the carboxyl-groups titrate quantitatively. The effect of acetone upon the basicity of the basic groups of amino-acids to phenolphthalein is similar to that of alcohol. The amino-acids tested up to the present have all given practically quantitative results when titrated with $\frac{N}{10}$ alkali in aqueous acetone containing 80 to 85 per cent. acetone. It has been shown further that the method is capable of much wider application. The total acids (including the carboxyl-groups of amino-acids), whether in the free state or combined with organic bases, can be accurately estimated in alcoholic preparations made from aqueous fluids such as those which contain the products of bacterial growth. As the titration value is unaffected by the free bases their removal is unnecessary. Consequently the solutions need no heating, and there is no risk of decomposition. A rapid method for estimating volatile bases is also described. H. F. E. H.

Rapid Estimation of Carbon. L. Lescœur. (*J. Pharm. Chim.*, 1920, 21, 267-263.)—A quantity of the substance (*e.g.*, sugar, organic acid, urine, etc.) containing .05 to .2 gm. of carbon is treated in a silver crucible with 10 grms. of a mixture of equal weights of sodium and potassium nitrates, and 25 c.c. of 2*N* sodium hydroxide solution (free from carbonate) are added; the whole mixture is evaporated to dryness and heated until fused. After cooling, the mass is dissolved in water, calcium chloride solution containing an excess of ammonium chloride is added, the precipitated calcium carbonate is collected after fifteen minutes, washed, and titrated with $\frac{N}{1}$ hydrochloric acid. W. P. S.

Volumetric Estimation of $\beta\beta$ -Dichloroethyl Sulphide. W. F. Hollely. (*J. Chem. Soc.*, 1920, 117, 898-902.)—The usual procedure for the estimation of $\beta\beta$ -dichloroethyl sulphide in "mustard gas" is to distil a known volume of the sample under diminished pressure and to collect the fraction boiling at 125-130° C./40 mm. This product is fairly pure $\beta\beta$ -dichloroethyl sulphide, melting at about 10°, but the procedure involves personal risks, and gives no accurate figure for the absolute percentage of $\beta\beta$ -dichloroethyl sulphide, as the latter fraction also contains higher chlorinated compounds and other impurities. The following volumetric method for the estimation of dichloroethyl sulphide in "mustard gas," with or without solvents, has, therefore, been devised, depending on the fact that $\beta\beta$ -dichloroethyl sulphide forms a definite double salt with cuprous chloride, of the constitution, $[(CH_2Cl.CH_2)_2S]_2 Cu_2Cl_2$. As the higher chlorinated compounds do not react with cuprous chloride, the method is applicable for the absolute estimation of $\beta\beta$ -dichloroethyl sulphide. About 1 gm. of the sample is weighed into a stoppered 100 c.c. conical flask, and to this 10 c.c. of a standardised solution of cuprous chloride in absolute alcoholic hydrogen chloride are added from a burette. The cuprous chloride solution should only be prepared immediately before use. It is best to have a 10 per cent. solution of hydrogen chloride in absolute alcohol on hand, and to dissolve 5 grms.

of pure cuprous chloride in 50 c.c. of it when required. The "mustard gas" sample readily dissolves in the alcoholic solution, and the contents of the flask are allowed to remain for ten minutes in the cold, with occasional rotation. At the end of this time the whole is gradually diluted with careful agitation and cooling in water, by the addition of 50 c.c. of a 5 per cent. aqueous sodium chloride. On total dilution the contents of the flask are well mixed by careful shaking, allowed to remain for a minute or two, and the liquid is then filtered from the precipitate through glass wool into a dry burette. The filtrate is quite clear, and the amount of copper in a known volume of it is subsequently determined in terms of c.c. of $\frac{N}{10}$ sodium thiosulphate, by the titration of iodine liberated from potassium iodide. It is impracticable to wash the double salt free from the excess of cuprous chloride, as it dissociates to some extent in contact with water. This difficulty is overcome by knowing the total volume of the 10 c.c. of standard cuprous chloride solution, plus the 50 c.c. of diluent, which is found to be 59.5 c.c. The total excess of cuprous chloride is thus readily calculated from the amount contained in the known volume of the filtrate taken for the copper estimation. Usually 30 c.c. of the filtrate are run from the burette into a 250 c.c. conical flask, and 5 c.c. of hydrogen peroxide (20 per cent. by volume) are added while cold to oxidise the copper to the cupric form. The contents of the flask are then gently boiled nearly to dryness, which operation is repeated twice after the addition of 10 c.c. of water to ensure that traces of gaseous oxidising agents are removed. The residue is diluted with 50 c.c. of water and sodium carbonate solution added to give a slight precipitate, which is redissolved by the careful addition of dilute acetic acid. A slight excess of potassium iodide is added, and the liberated iodine is titrated in the usual way with $\frac{N}{10}$ -sodium thiosulphate. Together with this, the original alcoholic cuprous chloride solution is standardised in terms of c.c. of $\frac{N}{10}$ -thiosulphate by oxidising 5 or 10 c.c. with hydrogen peroxide, and subsequently treating as already detailed. From the results of these titrations, the percentage of $\beta\beta$ -dichloroethyl sulphide is readily determined. The formula of the double salt being $[(CH_2Cl.CH_2)_2S]_2Cu_2Cl_2$, 127 grms. of copper correspond with 318 grms. of $\beta\beta$ -dichloroethyl sulphide. As 1 c.c. of $\frac{N}{10}$ -thiosulphate corresponds with 0.00635 grm. of copper, therefore 1 c.c. of $\frac{N}{10}$ -thiosulphate = 0.0159 grm. of $\beta\beta$ -dichloroethyl sulphide, the percentage of which is $\frac{(A - B) \times 0.0159 \times 100}{\text{weight of sample}}$, where A = c.c. of $\frac{N}{10}$ -thiosulphate corresponding with copper in the 10 c.c. of standard cuprous chloride solution, and B = c.c. of $\frac{N}{10}$ -thiosulphate corresponding with copper in the 59.5 c.c. of filtrate, that is, the total excess of copper.

H. F. E. H.

Preparation of Diphenylamine-Sulphuric Acid Reagent. F. Haun.

(*Zeitsch. Untersuch. Nahr. Genussm.*, 1920, **39**, 355-356.)—Occasionally diphenylamine reagent prepared with sulphuric acid free from nitric acid exhibits a blue coloration; this is caused by the presence of ferric salts in the sulphuric acid. Such acid may be rendered serviceable by heating it to boiling and allowing it to cool slowly; during the heating the ferric salts are reduced to ferrous salts, which do not interfere. If a small quantity of permanganate is added to the acid previously, the course of the reduction may be followed during the heating, since the permanganate is also reduced,

and a colourless acid results. The presence of the manganese salt does not interfere with the use of the reagent for the detection of nitric acid, and it even appears to increase the sensitiveness of the test.

W. P. S.

Estimation of Halogen in Organic Compounds. F. B. Dains and R. Q. Brewster. (*J. Amer. Chem. Soc.*, 1920, **42**, 1573-1579.)—The reaction between liquid ammonia, sodium, and organic compounds leads to the formation of cyanide in the case of a few substances only (chloroform, bromoform, carbon tetrachloride, chloral and bromal hydrates, ethylidene chloride, tetrachloroethylene, acetylene tetrachloride, methyl and benzyl cyanides, and ethyl cyanoacetate); the quantity of cyanide formed is variable, and the modification adopted by Clifford (*ANALYST*, 1919, **44**, 322) for its removal in the estimation of halogen is trustworthy. Application of the method to the estimation of cyanogen in organic compounds did not meet with success.

W. P. S.

Estimation of Hydrocyanic Acid. R. L. Morris. (*Pharm. J.*, 1920, **105**, 83-85.)—Comparison of various volumetric methods for the estimation of hydrocyanic acid showed that Liebig's method for the titration of cyanides is trustworthy as applied to hydrocyanic acid, provided that an excess of sodium hydroxide is not used for the preliminary neutralisation of the acid. A modification suggested by Guerin (*ANALYST*, 1906, **31**, 27), in which the hydrocyanic acid is neutralised by borax, has the advantage that an excess of borax does not interfere. Vollard's method is trustworthy, as is also that proposed by Fordos and Gelis (*Cf. Guerin, loc. cit.*). Mohr's method (titration with silver nitrate solution, using potassium chromate as indicator) has no advantage over that of Liebig. A modification of Liebig's method, proposed by Deniges, depends on the titration of the cyanide in strongly alkaline solution with silver nitrite solution, using potassium iodide as indicator; this method is accurate, and the B.P. (1914) method is based on it, but it appears that four times the quantity of potassium iodide prescribed is required.

W. P. S.

Specific Rotation of Lævulose. W. C. Vosburgh. (*J. Amer. Chem. Soc.*, 1920, **42**, 1696-1704.)—The specific rotation of lævulose at 25° C. can be expressed in terms of the per cent. by weight, p , and the concentration in grms. per 100 c.c., c , respectively, by the equations—

$$[\alpha]_{D 25^{\circ}} = -(88.50 + 0.145p) \text{ and}$$

$$[\alpha]_{D 25^{\circ}} = -(88.50 + 0.150c - 0.00086c^2).$$

These equations are based on experimental results, for the range $p = 2.6$ to $p = 18.6$, and $c = 2.5$ to $c = 20$. The temperature coefficient of the specific rotation is a function of the concentration; the relation between specific rotation and temperature for temperatures between 15° and 37° C. can be expressed as—

$$[\alpha]_{D t} = [\alpha]_{D 25^{\circ}} + (0.566 + 0.0028c)(t - 25).$$

W. P. S.

Determination of the Relative Strengths of some Nitrogen Bases of the Aromatic Series and of some Alkaloids. F. Arnall. (*J. Chem. Soc.*, 1920, 117, 835-839.)—The bases of the aromatic series are, on the whole, feebly basic, and are usually almost insoluble in water; the salts of these bases are, however, soluble. The author, therefore, measures the degree of hydrolysis of the hydrochlorides in aqueous solution by observing the acceleration of the rate of inversion of a solution of cane sugar by means of the polarimeter. The preparation of the hydrochlorides of many of these bases not being possible, their solutions have to be prepared with the addition of the calculated quantity of acid to the weighed base after careful purification of the latter. Equal volumes of the cane-sugar solutions (5 grms. per 100 c.c.) and the solution of the hydrochloride are brought to 55° C. in the thermostat, mixed and rapidly transferred to a jacketed polarimeter tube, care being taken to keep the temperature exactly constant; readings are taken at intervals, and the "final rotation," when the sugar was completely inverted, was read after the solution had been heated to 80° C. for fifteen minutes. The degree of hydrolysis of the hydrochloride is then deduced from the reaction velocity by comparison with the velocity found by hydrochloric acid alone. The degree of hydrolysis having been found at several dilutions, the dissociation constant of the salt, the basic constant of the base, and the relative strength of the base to aniline were then calculated. The following example of a typical case shows the results obtained:

Salt.	Dilution = V.	Degree of Hydrolysis	Dissociation Constant.
		$=\beta$ at 55° C.	$K_s = \frac{\beta^2}{(1-\beta)V^2}$
Dimethylaniline hydrochloride	4	0.0098	2.4×10^{-5}
	8	0.0133	2.3×10^{-5}
	16	0.0189	2.3×10^{-5}

The more common alkaloids, such as strychnine, brucine, cocaine, and morphine, were found to be too strongly basic to give satisfactory results by this method. Full tables are given showing the relative strengths and basic constants of twenty bases, of which the three following may be taken as representative:

RELATIVE STRENGTHS AND BASIC CONSTANTS OF THE BASES.

Base.	K_s .	$K_b = \frac{K_w}{K_s}$.	Aniline = 100.
<i>p</i> -Toluidine	4.0×10^{-5}	3.00×10^{-10}	250.00
<i>o</i> -Toluidine	1.59×10^{-4}	7.50×10^{-11}	62.00
Benzanilide	5.5×10^{-3}	2.18×10^{-13}	0.18

K_s = hydrolysis constant of the hydrochloric in aqueous solution.

K_b = basic constant.

K_w = dissociation constant for water, assumed to be equal to 1.2×10^{-14} , at 55° C.

It will be noted that all the values are relative to aniline, which is taken as 100. In another table is given the percentage degree of hydrolysis of hydrochlorides of some thirty bases, for which values the original paper should be consulted.

H. F. E. H.

Detection of Minute Quantities of Petroleum Spirit in Vegetable Oils.

M. Aida. (*J. Soc. Chem. Ind.*, 1920, 37, 152r.)—Nastjukoff's formolite reaction (*ANALYST*, 1912, 39, 274) may be applied successfully for the purpose as follows: The oil (50 to 100 grms.) is saponified by means of potassium hydroxide solution. Distilled water and pure calcium chloride solution are added, the liquid distilled by means of steam, and the distillate treated with 40 per cent. formaldehyde solution and a few drops of concentrated sulphuric acid. A reddish-brown film coloration on the surface of the liquid, gradually changing to deep yellow, indicates the presence of petroleum spirit. If a few drops of the distillate are added to water a brilliant interference ring of optical waves is produced on the surface of the water; this ring becomes almost invisible after standing for some time, and disappears completely on heating. With soya bean oil the ring does not change, even on heating. The above process is capable of detecting traces of petroleum spirit in vegetable oils, and may be made the basis of a quantitative method, the formolite precipitate being weighed after drying at 110° to 115° C. (see also *Proceedings Seventh Congress Applied Chemistry*, 1909, Section iv. A.1, 9-13).

H. F. E. H.

Determination of Phenol in the Presence of Certain other Phenols.

R. M. Chapin. (*J. Ind. and Eng. Chem.* 1920, 12, 771-775.)—The methods described are based upon the red colour produced by phenol with an accurately prepared Millon's reagent which, with many other phenols, yields yellow or orange colours. The reagent is prepared by dissolving 2 c.c. of mercury in 20 c.c. of concentrated nitric acid and diluting with 35 c.c. of water when, if any basic salt separates, nitric acid is added drop by drop until clear. Ten per cent. sodium hydroxide solution is then run in slowly with thorough mixing until the precipitate first formed no longer redissolves, and finally 5 c.c. of 0.2 per cent. (by volume) nitric acid are added. This solution should not be used when more than one day old.

Four c.c. of a neutral aqueous solution of the sample containing 0.1 per cent. of total phenols are measured into each of two test tubes, and to one of them is added about one-half the quantity of phenol believed to be present, both being then diluted to 6 c.c. Five c.c. of Millon's reagent are run in, the contents mixed, and the tubes placed in a boiling water-bath for exactly thirty minutes, afterwards being cooled in a current of water for ten minutes. Five c.c. of 0.2 per cent. nitric acid free from oxides of nitrogen are added, and the volumes made up to 25 c.c. by the addition of water. After mixing and standing for ten minutes the solutions are filtered until brilliant, transferred to the colorimeter, and the intensities of colour matched. The percentage of phenol present in the sample is calculated by means of a somewhat complicated mathematical formula, for which the original paper should be consulted. Several variations of the above method are described, which apparently yield equally satisfactory results.

The method is fairly accurate, and a minimum of 0.04 mgrm. of phenol in the presence of 4 mgrm. of cresol, etc., in a volume of 4 c.c. may be determined. It is applicable to a number of commercial products after simple preliminary treatment.

T. J. W.

Rapid Method of Analysing Phenolsulphonic Acids. L. Desvergnès. (*Ann. Chim. anal.*, 1920, 2, 211-214.)—From 5 to 6 grms. of the sample are dissolved in 200 c.c. of water, heating being avoided. Ten c.c. of the solution are diluted with 25 c.c. of water, boiled for fifteen minutes with 25 c.c. of hydrochloric acid saturated with bromine, diluted with 200 c.c. of water, and filtered from the insoluble bromine compound. The sulphuric acid is precipitated with barium chloride to obtain the total sulphuric acid present. The combined sulphuric acid is estimated by titrating 10 c.c. of the solution with $\frac{N}{2}$ sodium hydroxide with methyl orange as indicator, and deducting the result as sulphuric acid from the total sulphuric acid. For the estimation of the phenol 10 c.c. of the solution are treated with 100 c.c. of water, 50 c.c. of hydrochloric acid, and 25 c.c. of a standardised solution of 4 grms. of potassium bromate and 14 grms. of potassium bromide per litre, and the flask closed and heated, with occasional shaking, for thirty minutes on the water-bath at 50° C. The contents of the flask are then diluted with 100 c.c. of water, 5 c.c. of 20 per cent. potassium iodide solution added, and the liberated iodine titrated with sodium thiosulphate. This gives the amount of bromine which has not combined with the phenol. The amount of the latter is calculated by means of the equation—

$$\text{Phenol per cent.} = \frac{(B - bn) \times 200 \times 100 \times 94}{10 \times P \times 480} = \frac{(B - bn) \times 1,175}{3 P},$$

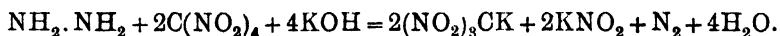
where P represents the weight of the sample, B the bromine in the 25 c.c. of bromate-bromide solution, and *b* the bromine equivalent of 1 c.c. of thiosulphate solution. The method gives accurate results with phenol sulphonic acids prepared with 93 per cent. sulphuric acid, but is less accurate when very concentrated acid is used, so that trisulphonic acid is present.

C. A. M.

Rapid Examination of Shellac, Shellac Varnish, and Lacquer. T. L. Crossley. (*J. Ind. and Eng. Chem.*, 1920, 12, 778-781.)—An approximate estimation of the amount of shellac in admixture with rosin, varnishes, etc., has been based upon the fact that the proportion of the resin insoluble in ether is fairly constant. Three samples of genuine shellac gum contained 70 to 70.2 per cent. of constituents insoluble and 28.7 to 31.8 per cent. soluble in ether, the discrepancy in one instance being probably due to oxidation. A sample of crude stick-lac gave the following results: Woody fibre, etc., 8.5; gums insoluble in ether, 70.7; and gums soluble in ether, 25.0 per cent. Mixtures of shellac and rosin in varnish examined by this method gave results agreeing closely with the manufacturers' formulæ: A weighed quantity (5 to 7 grms.) of varnish is thoroughly mixed with a large excess (50 to 60 c.c.) of ether in a tared beaker, the solution decanted through a filter into a second tared beaker, and the insoluble portion washed with successive portions of 20 c.c. of ether until the washings are practically colourless. If any deposit forms in the

filtered solution it is separated in a third beaker, and the solution is then evaporated and the residue dried and weighed. The insoluble portions in the other beakers are also dried and weighed. The total gums in the varnish are found by evaporating 5 to 10 grms. of the original varnish, and the proportion of shellac is calculated on the basis of 70 per cent. of insoluble gums in the genuine resin. C. A. M.

Effect of Reducing Agents on Tetranitromethane, and a Rapid Method of Estimation. A. Baillie, A. K. Macbeth, and N. I. Maxwell. (*J. Chem. Soc.*, 1920, 117, 880-884.)—The paper describes a study of alkaline reduction methods as applied to the estimation of tetranitromethane. These reductions result in the removal of the labile "nitroite" group with the production of salts of nitroform, which may be conveniently and safely prepared by the methods described. The procedure consisted in agitating the nitro-compound with aqueous solutions of the reducing agents containing a requisite amount of potassium hydroxide, which neutralises the nitroform produced, precipitating it as the sparingly soluble potassium salt. The solutions are thereby prevented from becoming acid, and secondary action with the reducing agent is avoided. Aqueous potassium hydroxide itself attacks tetranitromethane, with the formation of the potassium salt of nitroform, together with nitrate, nitrite, and carbonate of potassium, the quantities of which vary with concentration. The decomposition in such case, however, is much slower than when a reducing agent is present, and in the latter case the tetranitromethane is generally all converted into nitroform without the loss entailed in the formation of potassium carbonate. Results have been obtained, amongst others, with sodium sulphite, potassium sulphite, sodium potassium tartrate, and hydrazine. In all cases the nitroform salt has been identified by estimation of the percentage of potassium contained in the salt recrystallised from water; this was decomposed by sulphuric acid and the potassium weighed as sulphate. The reagent finally adopted was hydrazine from which the reaction proceeds according to the equation—



This gives results within the limits of experimental error. Half a gram of tetranitromethane is a convenient amount to use, as it liberates some 30 c.c. of nitrogen from hydrazine solutions. As the estimation is carried out in a Lunge nitrometer graduated to 0.2 c.c. the experimental error in reading the volume of gas evolved in the reaction may well be this amount. From the quantitative relation given above, 0.2 c.c. of nitrogen corresponds approximately with 0.0035 grm. of tetranitromethane. Using 0.5 grm. of the nitro-compound, the error in reading the nitrometer should theoretically be within a margin of 1 per cent. The results obtained are well within this limit. Numerous precautionary details are described and it is found that the main reaction is over in less than a minute, but further traces of nitrogen are evolved on shaking once to mix the solutions thoroughly. As the reaction is exothermic the nitrometer must be allowed to remain for some time before a volume reading is taken. Sodium hydroxide may be substituted for potash, but the latter is preferable. Conversely, standard solutions of tetranitromethane may be employed in the estimation of hydrazine in solutions of its salts. H. F. E. H.

Colorimetric Estimation of Tyrosine by the Method of Folin and Denis. R. A. Gortner and G. E. Holm. (*J. Amer. Chem. Soc.*, 1920, **42**, 1678-1692.)—Previous workers having found the above method (see *J. Biol. Chem.*, 1912, **12**, 245) unsatisfactory, the authors describe numerous experiments performed in order to determine the reliability of the results obtained by it. The method is found to be unreliable unless it is certain that no substance other than tyrosine is present which is capable of yielding a blue colour with the phenol reagent used. This reaction is also given by tryptophane, indole and its derivatives, various other substances produced by the hydrolysis of proteins, ferrous iron, and any other easily oxidised substances. Uncertainty in the results is also due to the colour development not being a linear function of the amount of reactive material present, and it is necessary to know the approximate concentration of the solution beforehand so that the maximum colour will be developed. Bone-black cannot be used for decolourising protein hydrolysates when employing this method, for not only does this material absorb tyrosine in appreciable amounts, but it yields to an acid solution some easily oxidisable substance which yields a blue colour on addition of the reagent. On the whole the method has little to recommend its adoption. T. J. W.

Detection of Water in Alcohol and other Organic Solvents. F. Henle. (*Ber.*, 1920, **53**, 719-722, through *J. Soc. Chem. Ind.*, 1920, **39**, 501A.)—A xylene solution of partially decomposed aluminium ethoxide is prepared by allowing aluminium turnings (27 grms.), absolute alcohol (276 grms.) and mercuric chloride (0.2 grms.) to react until the evolution of hydrogen subsides, and then heating the product on the water-bath until it appears dry. The alcohol of crystallisation is then distilled from an oil-bath at 210° to 220° C., and the crude aluminium ethoxide is subsequently cautiously heated until the temperature recorded by a thermometer immersed in the molten mass sinks from 340° C. to about 330° C. The product, whilst still hot, is dissolved in 500 c.c. of boiling xylene, and the solution is filtered through a dry paper; the clear, pale yellowish-brown filtrate can be preserved indefinitely if air and moisture are excluded. If a few drops of the reagent be added to a few c.c. of the liquid under examination, then, according to the quantity of water present, a voluminous, gelatinous precipitate of aluminium hydroxide is formed immediately or in the course of a few seconds. The percentage of water which can thus be detected in various solvents is as follows: ethyl alcohol, 0.05; methyl alcohol, 0.1; ether, 0.005; ethyl acetate, 0.1; acetaldehyde, 0.1; acetone, 1. The two substances last named also yield white turbidities due to the precipitation of aluminium ethoxide from the reagent, but these can be readily distinguished from aluminium hydroxide, since they immediately disappear on addition of a larger excess of reagent or of xylene.

INORGANIC ANALYSIS.

Preparation of Water free from Ammonia. G. C. Baker. (*J. Ind. and Eng. Chem.*, 1920, **12**, 798-799.)—Distilled water may be freed from ammonia (but not from nitrate, nitrite, or albuminoid nitrogen) by filtration through permutit. An automatic feeding apparatus has been devised for the purpose, consisting of a large

glass aspirator bottle containing a layer of glass beads 1.5 inches deep, covered with 12 to 14 inches of permutite. The distilled water enters the bottle at the top, and is distributed by a perforated glass bulb. It is filtered at the rate of about 1 gallon per hour, and rises from the outlet tube at the bottom through an outside tube almost to the height of the level of the water inside, and is then delivered downwards, the bent tube having an opening at the top to prevent it acting as a siphon. An overflow pipe is provided for use in flushing the filter backwards. The permutite is regenerated by passing 2 gallons of a 10 per cent. solution of common salt on to the filter, allowing it to stand overnight, and then washing the bed. It has not been determined whether the removal of the ammonia is solely a displacement of the sodium in the hydrated sodium aluminium silicate ($\text{Na}_2\text{Al}_2\text{Si}_4\text{O}_{12}\cdot 6\text{H}_2\text{O}$) or whether there is also mechanical absorption of the ammonia.

C. A. M.

Electrometric Analysis of Arsenic Compounds. C. S. Robinson and O. B. Winter. (*J. Ind. and Eng. Chem.*, 1920, 12, 775-778.)—Electrometric methods of estimating arsenic are useful in the case of trivalent arsenic in coloured solutions, and for the direct estimation of pentavalent arsenic, without removal of organic matter or preliminary reduction to the trivalent condition. The apparatus described is essentially the same as that used by Hildebrand (*J. Amer. Chem. Soc.*, 1913, 35, 869), with the addition of a mechanical stirrer, and the substitution of a reflecting galvanometer for the capillary electrometer. The titrations are effected in a beaker, into which dip a platinum electrode and a calomel half-element. In estimating trivalent arsenic in, e.g., London purple, 4 grms. of the sample are dissolved in hydrochloric acid below 60° C., the solution made up to 500 c.c., rendered alkaline with sodium bicarbonate, the sliding contact set to indicate the proper voltage (e.g., about 260 m.v.) on the voltmeter, and the liquid mechanically stirred and titrated with $\frac{N}{10}$ iodine solution, until the completion of the oxidation is indicated on the galvanometer. For the estimation of pentavalent arsenic, the titration with hydriodic acid must be carried out in a strongly acid solution (50 per cent. by volume of sulphuric acid at the end of the titration) and at about 90° C. A special burette is used, the tip being drawn out to a capillary and bent upwards to form a hook, thus allowing the solution to be introduced at the bottom of the beaker, and to rise through the liquid before reaching the air. The reagent must not be added too rapidly, since the voltmeter reading changes slightly after the addition. Under the conditions described, good results were obtained by setting the sliding contact so that the millivoltmeter gave a reading of about 350 m.v., and titrating the solution until the galvanometer showed no movement or just reversed the direction.

C. A. M.

Iodimetric Estimation of Arsenic Acid. P. Fleury. (*J. Pharm. Chim.*, 1920, 21, 385-391.)—The arsenic acid is reduced by hydriodic acid to arsenious acid, and the latter is titrated with iodine solution. A quantity of the arsenate, equivalent to about 50 c.c. of $\frac{N}{10}$ iodine solution (e.g., 0.7 gm. of sodium arsenate), is dissolved in not less than 30 c.c. of water, 1 c.c. of hydrochloric acid (sp. gr. 1.171) is added for each 10 c.c. of the solution, the mixture is heated on a boiling water-bath

for five minutes, potassium iodide is then added in quantity sufficient to make a 25 per cent. solution, the heating is continued for a further ten minutes, the mixture then cooled, and the liberated iodine eliminated by the cautious addition of dilute thiosulphate solution. An excess of sodium hydrogen carbonate is now added and the solution titrated with $\frac{N}{10}$ iodine solution. Direct titration of the iodine, liberated by the reaction, yields high results, owing to the action of atmospheric oxygen on the hydriodic acid, therefore this free iodine is destroyed by the addition of the requisite amount of thiosulphate, and the arsenious acid then estimated iodimetrically.

W. P. S.

Composition of Ancient Eastern Bronzes. M. Chikashige. (*J. Chem. Soc.*, 1920, 117, 917-922.)—The paper deals with the analysis of ancient Japanese and Chinese bronzes. The author's method in the case of corroded objects is to remove the earthy matter from a rusty article and to analyse rust and metal together. This gives, of course, too low a percentage for the metallic constituents, which should therefore be recalculated for 100. Even this method becomes doubtful where the composition of the rust has undergone change by a partial solution or disintegration before analysis. Four classes of ancient bronze mirrors were examined—namely, white, pale yellow, deep yellow, and red, according to their surface colours. These colour differences are due to the amounts of tin becoming less and less, and correspond exactly with the chronological period in which the mirrors were made. In the most recent times zinc is employed in place of tin, giving rise to the use of brass mirrors. All but the white and pale yellow had necessarily to be alloyed with mercury before use. A table summarises the analytical results obtained in the examination of sixteen specimens of Chinese, Japanese, and Korean bronze mirrors. The copper to tin ratio in most of them runs about 70 to 30, the extreme values found for copper were from 58 to 74 per cent., tin from 3.3 to 30.7 per cent., lead from 4 to 20.5 per cent.; small quantities of zinc, iron, arsenic, antimony, nickel, and, in one case, silver and gold, were also found. The presence of lead is not to be regarded as useless since it reacts with neither copper nor tin, but lies between the granules of the solid solution, acting as a cement. Without lead the mixture is apt to be too brittle to be polished. Analyses of swords, halberds, and arrow-heads are included, and numerous analyses of bell metal, money, and spoons are also recorded. Ancient Chinese coins generally contain less tin with much lead, so that they can scarcely be called bronze. Some Chinese money made between 700 and 500 B.C. contained copper running from 38 to 70 per cent., tin from 1.6 to 16 per cent., lead from 10 to 55 per cent., with the usual small quantities of antimony, iron, arsenic, and nickel. Bell metal with a high percentage of tin is sonorous, but too brittle. A Japanese bell dated about the eighth century, A.D., contained 69 per cent. copper, 15.5 per cent. tin, 5.6 per cent. lead, 8.3 per cent. antimony, 1.35 per cent. nickel, and traces of iron and arsenic.

H. F. E. H.

Volumetric Estimation of Hydrosulphides in the Presence of Sulphides, Thiosulphates, and Sulphites. A. Wöber. (*Chem. Zeit.*, 1920, 44, 601.)—Sodium hydrosulphide, when treated with an excess of mercuric chloride solution, reacts

according to the equation $2\text{NaHS} + 3\text{HgCl}_2 = 2\text{NaCl} + 2\text{HCl} + (2\text{HgS} \cdot \text{HgCl}_2)$; thio-sulphate reacts similarly, with the liberation of 2 molecules of sulphuric acid (*Cf.* Sander, *ANALYST*, 1916, 41, 84), whilst sulphide and sulphite give neutral solutions. If a portion of the solution containing the four salts is mixed with mercuric solution and the acidity produced titrated after the addition of ammonium chloride, using methyl-orange as indicator, the acidity found is a measure of the hydrosulphide and thiosulphate content (*A*). Another portion of the solution is then treated with zinc carbonate to remove hydrosulphide and sulphide, filtered, and the thiosulphate estimated alone as just described. The difference between the acidity thus found *B*, and *A* gives that due to the hydrosulphide (*X*). To estimate sulphide and sulphite a measured quantity of the solution is added to a definite quantity of $\frac{N}{10}$ iodine solution acidified with 10 c.c. of $\frac{N}{10}$ hydrochloric acid, and the excess of iodine is titrated with $\frac{N}{10}$ thiosulphate solution; the quantity of iodine used is a measure of the sulphide, hydrosulphide, thiosulphate, and sulphite (*C*); the acidity of the mixture is then titrated with $\frac{N}{10}$ sodium hydroxide solution, using methyl-orange as indicator. Hydrosulphide and sulphite form hydriodic acid when treated with iodine according to the equations $\text{NaHS} + \text{I}_2 = \text{NaI} + \text{HI} + \text{S}$ and $\text{Na}_2\text{SO}_3 + \text{I}_2 + \text{H}_2\text{O} = \text{Na}_2\text{SO}_4 + 2\text{HI}$. After deducting the quantity of added hydrochloric acid, the remaining acidity is due to hydrosulphide and sulphite (*D*), therefore the sulphite (*y*) is equivalent to $D - x$. The results so far obtained allow the sulphide (*z*) to be calculated by the formula $C - (2x + B/2 + y) = z$.

W. P. S.

Iodic Acid as a Characteristic Microchemical Reagent for Gaseous Ammonia. G. Denigès. (*Comptes rend.*, 1920, 171, 177-179.)—Aqueous solutions of ammonium salts only yield crystalline precipitates with iodic acid when at least 15 to 20 per cent. of the salt is present. With gaseous ammonia, however, flat rectangular crystals of ammonium iodate, NH_4IO_3 , are immediately produced. A minute drop of a 10 per cent. solution of iodic acid, when exposed for a few seconds to ammonia vapour, becomes coated with a layer of these crystals, which may be recognised under the microscope and by their action on polarised light. The reaction is distinctive, not being given by any volatile amine, and is also very sensitive. In using it for the identification of ammonium salts in water, etc., the liquid is evaporated to about 1 c.c., and introduced, together with 0.5 gm. of calcined magnesia, into a short glass tube (3 to 4 cm. long and 15 to 16 mm. in diameter), over the mouth of which is placed an object-glass with a suspended drop of the iodic acid solution coinciding with the axis of the tube. If 1 mgrm. of ammonium salts be present, crystals will appear in a few minutes; in less than thirty minutes with 0.2 mgrm.; and in about an hour with 0.1 mgrm.

C. A. M.

Estimation of Lead in Acid Solution. N. Evers. (*Pharm. J.*, 1920, 105, 85-86.)—In cases where it is necessary to estimate lead colorimetrically in acid solution the test and comparison solution must have the same acidity, otherwise the colorations produced by hydrogen sulphide are not comparable. By using bromophenol blue as indicator, the solutions may be brought to the colourless point of the indicator by the addition of alkali or acid (*p* then equals about 3.8); when

hydrogen sulphide solution is added, the lead coloration may be matched against that of a standard prepared in the same way. Iron does not give a coloration with hydrogen sulphide under these conditions, and the interference of copper may be prevented by the addition of cyanide; if, however, iron is present with copper, complications arise owing to the formation of ferrocyanides. It is, therefore, preferable to remove the copper by adding alum and ammonia to the solution, collecting the precipitate of aluminium hydroxide (this contains the lead, whilst the copper remains in solution), redissolving it in acid and treating the solution as described.

W. P. S.

Separation of Magnesium from Sodium and Potassium Chlorides. S. Palkin. (*J. Amer. Chem. Soc.*, 1920, **42**, 1618-1621.)—A method, described previously by the author (*ANALYST*, 1917, **42**, 54) for the separation of sodium and potassium from lithium, may be applied to the separation of the two former from magnesium, since magnesium chloride is soluble in the alcohol-ether reagent. The procedure is the same, except that 25 c.c. of absolute alcohol and 25 c.c. of ether are used for the precipitation, and that the filtrate and washings from the precipitated sodium and potassium chlorides are evaporated to dryness, and the residue dissolved in alcohol-ether mixture; a further quantity of a few mgrms. of the alkali chlorides is thus recovered. After removing the alcohol and ether from the filtrate, the magnesium may be estimated by the phosphate method.

W. P. S.

Estimation of Nickel in Steels by Direct Titration. E. H. Hall. (*J. Soc. Chem. Ind.*, 1920, **39**, 253-254r.)—The ordinary method of titrating nickel by means of potassium cyanide in a tartaric (or citric) acid solution of the steel has the drawbacks that it is difficult to observe the exact point when the final turbidity is formed, and that the excess of ammonia added is not controlled. The first drawback is obviated by transferring the tartaric acid solution of the steel, before complete neutralisation, to a measuring cylinder, which is supported over a mirror adjusted to reflect light upwards through the column of liquid. The other difficulty is obviated by taking advantage of the fact that silver chloride is soluble in ammonia solution, and that as soon as free ammonia is present any cloudiness due to the chloride will disappear. The following modification of the method gives results agreeing within the second place of decimals: The solution of 0.5 gm. of the steel in 10 c.c. of concentrated hydrochloric acid is oxidised with 2 c.c. of nitric acid on the water-bath, diluted with 35 c.c. of hot water, vigorously boiled on a hot-plate to expel nitrous fumes, and treated first with 12 c.c. of tartaric acid solution (100 grms. in 200 c.c.), and then with 8 c.c. of ammonia solution (sp. gr. 0.880). After cooling, the liquid is transferred to a Nessler cylinder holding at least 150 c.c., and about 2 c.c. of standard silver nitrate solution (2 grms. per litre) run in from a burette. The liquid is then titrated with dilute ammonia solution (sp. gr. 0.956), and stirred with a glass plunger until the turbidity due to the silver chloride disappears, this point indicating neutrality. An excess of 1 c.c. of the ammonia solution is then added, and on now adding 2 c.c. of 2 per cent. potassium iodide solution a turbidity of silver iodide is produced. Finally, the liquid is titrated with potassium cyanide solution (6.7 grms.

KCN and 1 grm. KOH per litre), a slight excess being added, and this excess titrated with the standard silver nitrate solution. In calculating the percentage of nickel the original 2 c.c. of silver nitrate solution is included. The potassium cyanide solution is best standardised upon a steel containing a known quantity of nickel.

C. A. M.

Simultaneous Precipitation of Copper and Nickel Oxides with Ferric Hydroxide. E. Torporescu. (*Comptes rend.*, 1920, 171, 303-305.)—Ferric hydroxide was precipitated in the cold by means of ammonia from solutions containing 0.2948 per cent. of ferric chloride with variable quantities of solutions of 0.3692 per cent. of copper sulphate and 0.611 per cent. of nickel chloride, and the precipitates left for three hours in contact with the liquid. The amount of copper oxide or of nickel oxide in the ignited precipitate increased with the concentration of the second salt when that of the other was kept constant. For example, the precipitates contained from 11.4 to 37.1 per cent. of copper oxide. It was not possible to free the ferric oxide completely from either copper oxide or nickel oxide by washing with ammonium nitrate or ammonia solutions, or by a second precipitation with ammonia.

C. A. M.

Estimation of Phosphoric Acid in Phosphates of Heavy Metals. F. Seeligmann. (*Chem. Zeit.*, 1920, 44, 599.)—Ten grms. of the finely powdered phosphate are mixed with about 12 grms. of sodium hydroxide solution (sp. gr. 1.075), and heated to 90° to 95° C., with continual stirring. After about fifteen minutes the solution is diluted with 3 to 4 times its volume of water, boiled, and made up to a litre. An aliquot portion is filtered, rendered slightly acid with acetic acid, and the phosphoric acid titrated with uranyl acetate in the usual way. Results in close agreement with those given by the molybdate method have been obtained with iron, zinc, and aluminium phosphates.

C. A. M.

Estimation of Potassium by the Cobaltinitrite Method. P. Wenger and C. Hémen. (*Ann. Chim. anal.*, 1920, 2, 198-199.)—Inaccuracies in the ordinary methods of estimating potassium by precipitation as sodium potassium cobaltinitrite are obviated by estimating the cobalt in the precipitate and calculating it into the corresponding proportion of potassium in the salt $\text{NaK}_2\text{CO}(\text{NO}_2)_6$. The precipitate obtained by the usual method is washed and dissolved in hydrochloric acid, and the cobalt precipitated by means of sodium hydroxide, and weighed as metallic cobalt. Or, the precipitate may be treated with sulphuric or hydrochloric acid, and the resulting solution of cobalt sulphate or chloride electrolysed. The weight of the cobalt estimated by either method, multiplied by 1.5932, gives the corresponding quantity of potassium oxide.

C. A. M.

Analysis of Sodium Silicofluoride. P. Drawe. (*Zeitsch. angew. Chem.*, 1919, 33, 312; *Chem. Zeit. Uebers.*, 1920, 44, 201.)—Commercial sodium silicofluoride, at the present time, frequently consists of mixtures of sodium silicofluoride, sodium silicate, and hydrated silicic acid. The amount of silicofluoride may be

estimated by treating 0.6 to 1 gram. of the sample with an excess of $\frac{N}{4}$ hydrochloric acid, diluting the mixture with water, and exactly neutralising the solution (to methyl orange) with $\frac{N}{4}$ potassium hydroxide solution. The neutral solution is then treated with a few drops of phenolphthalein, heated, and titrated with $\frac{N}{4}$ potassium hydroxide solution: $\text{Na}_2\text{SiF}_6 + 4\text{KOH} = 2\text{NaF} + 4\text{KF} + \text{Si}(\text{OH})_4$.

C. A. M.

Volumetric Estimation of Sulphate Ion. J. Erlich. (*Ann. Chim. anal.*, 1920, 2, 214, 215.)—The solution, which should contain about 0.01 gram. of sulphuric acid entirely in the form of an alkali salt, is exactly neutralised with sodium hydroxide or dilute hydrochloric acid, diluted to 300 to 350 c.c., boiled with an excess (about 1 gram.) of pure powdered barium carbonate, and filtered. The precipitated barium sulphate is washed three or four times with boiling water, and the alkali carbonate in the filtrate titrated with $\frac{N}{10}$ sulphuric acid.

C. A. M.

Estimation of Sulphate in Sulphonated Oils. E. J. Kern. (*J. Ind. and Eng. Chem.*, 1920, 12, 785.)—Free sulphates may be extracted from an oil by means of one extraction with 10 per cent. mono-sodium phosphate solution as completely as by three extractions in the usual way with brine and ether. In estimating the total sulphuric acid in sulphonated oils, the sample may be boiled with hydrochloric acid to decompose the oil, and the sulphate extracted with the sodium phosphate solution. A better method is to evaporate the oil with sodium carbonate, to ignite and fuse the residue, to dissolve the mass in water, and to boil the solution with sodium peroxide. The liquid is then acidified, and the sulphate precipitated with barium chloride.

C. A. M.

Analysis of Commercial Zinc. E. Olivier. (*Ann. Chim. anal.*, 1920, 2, 199-207, 226-234.)—Methods, mainly colorimetric, are described for the estimation of the commoner impurities present in commercial zinc; standard specimens containing known amounts of the impurities are used for comparison. Tin is estimated by separation as metastannic acid and as its sulphide, iron by means of thiocyanate, and lead, copper, cadmium, antimony, and arsenic are estimated by their sulphide colorations.

W. P. S.

APPARATUS, ETC.

Alundum Filtering Crucibles. D. T. Englis. (*J. Ind. and Eng. Chem.*, 1920, 12, 799.)—The difficulty of removing all soluble salts, especially from the upper edge of these crucibles, is overcome by the author as follows: The crucible containing the precipitate and fitted into a conical glass funnel by means of a rubber flange, is half filled with water, the vacuum turned on, and a smooth rubber stopper placed firmly upon the top edge of the crucible. The surrounding funnel is then filled with water, which is rapidly drawn through the upper wall of the crucible, thus washing out all soluble matter.

T. J. W.

Gas Analysis Apparatus, Accurate to 0.001 per cent., mainly designed for Respiratory Exchange Work. A. Krogh. (*Biochem. J.*, 1920, 14, 267-281.) The paper deals in a very full and elaborate manner with the experimental methods employed in gas analysis, and is fully illustrated. By employing current methods, carbon dioxide can be determined with any desired accuracy (to 0.0005 per cent.), but to obtain accurate oxygen determinations is more difficult, and even for the best instruments in actual practice errors of 0.1 per cent. are common. Since the oxygen deficit in the air leaving a respiration chamber cannot be increased beyond 2 per cent., and for many reasons should not be increased beyond 1 per cent., the accuracy obtainable is scarcely satisfactory. A great deal would be gained if the accuracy of the gas analysis could be increased sufficiently to allow an oxygen deficit of 0.5 to 1 per cent. to be analysed without causing errors exceeding 0.2 per cent., and this the author endeavours to obtain. The principal sources of error preventing the oxygen analysis being as accurate as the CO₂ determinations, are intimately connected with the presence of water and dirt in the gas burette. Water must be present to insure the saturation of the gas with vapour, since it is found impracticable to measure this completely dry, while dirt will accumulate rather rapidly from the contact of the mercury with the rubber tubing and with oxygen. The two main improvements described by the author are, firstly, the employment of three separate gas burettes, and, secondly, the raising and lowering of the mercury in the burettes by means of air pressure, thus obviating the use of rubber connections between the burettes and the reservoir. The soda or potash employed for CO₂ absorption, moreover, should never be more concentrated than 10 per cent., and must also be saturated with air; while the pyrogallate solution should be saturated with nitrogen at ordinary barometric pressure and the temperature of the bath. For absolute determinations the pyrogallate solution must also be tested with regard to its absolute absorbing power and the formation of carbon monoxide. When the potassium hydroxide used to make up the pyrogallate solution is not concentrated, very appreciable amounts of carbon monoxide are produced by the absorption of oxygen, and if a control analysis of the standard air sample shows a deviation which is outside the limits of accidental error (+0.001 per cent. for the nitrogen) the experimental analyses made on the same day should be corrected accordingly. Numerous tables are given showing the order of accuracy obtainable, and very full working details—for which the original paper must be consulted—are also described.

H. F. E. H.

Calibration, Accuracy, and Use of Gas Meters. A. Krogh. (*Biochem. J.*, 1920, 14, 282-289.)—The gas meters described are those employed in connection with respiration-chamber work, a calibration spirometer with improvements for calibrating gas meters being fully described and illustrated. The paper also deals with wet gas meters and their accurate employment, as well as dry gas meters consisting of two bellows which are alternately filled and emptied by the current of air passing through. These require no filling, and will work equally well in any position. Wet gas meters properly handled are instruments of precision. Motor-driven wet meters acting as pumps, and, with a constant water level maintained by a slow current of

water, are accurate to less than 0.1 per cent. at all rates below 1 revolution per minute. When they are calibrated for more rapid rates they can be used with almost the same accuracy at rates up to 3 revolutions per minute. The volume per revolution decreases with increasing rate. In wet meters with a constant quantity of water the volume per revolution increases with increasing rate, but can be determined with equal accuracy. The subdivisions of the revolution of an experimental wet meter (Bohn meter) usually show slight errors which can be allowed for after calibration. Dry gas meters are, on the whole, less accurate than wet, and when they are arranged to show the volumes directly in litres their indications are often very inaccurate and cannot be calibrated. They should be arranged to count revolutions. Gas volumes representing fractions of a complete revolution are generally very inaccurately indicated, but the volume corresponding to a whole revolution is a practically constant quantity. Varying rates may cause variations in the volumes registered, but at practicable rates usually within 1 per cent. The calibration of a dry meter changes with age.

H. F. E. H.

Tests for Laboratory Resistance Glassware. (*J. and Proc. Inst. of Chem.*, 1920, Part III., 202-210.)—The Glass Research Committee of the Institute of Chemistry have prepared a scheme for testing laboratory glassware, with which the National Physical Laboratory have agreed generally, the object being to characterise such glassware as may safely be considered "resistance" glass. The tests, which are fully described in detail, are the minimum which such glass ought to answer, and no attempt is made to distinguish between different makes of resistance glass, nor are the tests intended to ascertain the extreme limits of resistance. For the protection of the user the described tests afford a simple means of ascertaining whether glass has any claim to be considered of resistance quality for ordinary chemical operations, while for toxicological or other special purposes the user must, of course, apply his own tests for specific purposes. The following is but a summary of the tests to be employed; for details the original must be consulted:

1. *Preliminary Treatment.*—Cleaning with boiling water, followed by 5 per cent. acetic acid.

2. *Treatment in Autoclave.*—This affords a rapid sorting test for resistance glass. The glass vessel, filled with distilled water, is heated for three hours in an autoclave at a registered pressure of 4 atmospheres. Any matter dissolved is then estimated by drying a portion of the solution for one hour at 120° C., followed by ignition (not above 650° C.) for three minutes. The result is expressed as mgrm. residue per sq. dm. The alkalinity of the rest of the water is determined by titration, using methyl orange. Express results as c.c. $\frac{N}{100}$ H_2SO_4 per square dm.

3. *Treatment with Reagents.*—Hydrochloric acid (sp. gr. 1.15) is boiled for half an hour in the vessel to be tested, the whole being enclosed in an air-bath kept at 140° C. This is repeated three times (each time with fresh acid), and finally the dissolved residue is dried down with a little ammonium carbonate, ignited, and weighed. Silica and zinc are both to be tested for in the residue; after the acid treatment the same vessel is tested by boiling in it $\frac{N}{2}$ ammonium chloride and ammonia, the dissolved matter, if any, being determined as before, all results being

expressed as mgrms. per sq. dm. Finally, $\frac{N}{2}$ sodium hydroxide is employed, the residue being tested for zinc and lead.

4. *Heat Tests—Alternative Methods.*—(a) The flask or beaker is heated in an air-oven to 120° C. for half an hour, and then instantly immersed in cold water. (b) The vessel is filled with soft paraffin wax and heated to about 155° C. and plunged in cold water; this is repeated at increasing temperatures (25° C. intervals) until it breaks. Good quality vessels will stand this treatment up to 200° C. (c) A solution of calcium chloride (sp. gr. 1.33) is boiled in the vessel for five minutes, and the whole then plunged into water at 0° C. (d) Another vessel full of water at 0° C. is plunged into boiling water.

Vessels should not crack when submitted to tests under (a), (c), and (d).

5. *Tests for Arsenic and Antimony.*—Full accounts of testing for these elements are given, with a diagram of the apparatus for distillation.

6. *Annealing.*—The vessels are examined under polarised light to detect interval stresses.

Convenient dimensions of vessels to be tested are given and the following maxima for resistance glass are recommended:

Autoclave Test.—Must not exceed 4 mgrms. per sq. dm. Alkalinity should not require more than 5 c.c. $\frac{N}{100}$ H_2SO_4 per sq. dm. The glass must not flake or peel.

Hydrochloric Acid Test on Evaporation to Dryness.—First treatment: Residue must not exceed 2 mgrms. per sq. dm. Second treatment: 1 mgrm. per sq. dm.

Ammonia and Ammonium Chloride Test.—Residue must not exceed 1.5 mgrm. per sq. dm.

H. F. E. H.

Tests for Laboratory Porcelain. (*J. and Proc. Inst. Chem.*, 1920, 210-214.)—

The tests described are those adopted by the sub-committee on porcelain of the Glass Research Committee of the Institute of Chemistry.

1. *Appearance, Shape, and Weight.*—These should at least come up to that of the porcelain in former use. The weights of vessels should not exceed pre-war average weights for articles of similar size.

2. *Tests for Porosity of Body and Imperfections in Glaze (Dye Test).*—A 0.5 per cent. solution of eosin in water answers well. Some vessels are filled and broken pieces of others immersed in the solution for eighteen hours, after which they are rinsed, dried, and examined with a hand lens. Good porcelain shows no staining at all. If the articles fail under this "dye" test, it is extremely unlikely that they will stand the remaining tests.

3. *Resistance to Heat and Sudden Changes of Temperature.*—(a) The vessels are heated by direct application of Bunsen burners, and then lifted with small, cold tongs on to cold pipeclay triangles to cool. This is repeated six times and the "dye" test again applied. (b) After thorough drying the same vessels are heated as before and cooled by being placed on cold metal (clean sheet lead). The "dye" test as before is then applied.

4. *Constancy of Weight and Resistance of Glaze to High Temperatures.*—(a) Vessels should suffer no loss of weight after heating for several hours at a good red heat and should show no tendency to stick to pipeclay, silica, or other supports. (b) The

condition of the glaze should be noted after heating for four hours at 950° C. No blistering or coaging should result.

5. *Cleaning Test*.—Cleaned, ignited, and weighed vessels should show no change in weight after immersion for twelve hours in dilute acid followed by rinsing, wiping, and ignition. Under this test, if the body is porous, liquid will have entered and on the application of sudden heat particles of glaze or porcelain will be thrown off by vapour generated from the imprisoned liquid, with resulting loss of weight.

6. *Resistance of Glaze to Acid and Alkali*.—The loss in weight sustained after prolonged treatment with boiling acid, carbonate of soda, and caustic soda should not exceed that found in comparison with high-grade pre-war porcelain.

Two simple tests for lead are finally given: (1) The porcelain is touched with hydrofluoric acid, warmed, and the acid allowed to evaporate, followed by sulphuretted hydrogen water faintly acidified with acid. (2) Fragments of the porcelain are heated to bright redness for four hours in a current of hydrogen. No darkening should ensue.

H. F. E. H.

Yellow Light in Polarimetric Determinations. L. Guglielmelli. (*Anal. Soc. Quim. Argentina*, 1918, 6, 497-499; through *J. Chem. Soc.*, 1920, 117, ii, 444.)—To obviate the drawbacks of the use of sodium chloride, Dupont proposed a mixture of salt and trisodium phosphate. This is found to work very satisfactorily. For its preparation 1 gram-molecule of disodium phosphate is dissolved in water and treated with 1 gram-molecule each of sodium hydroxide and chloride. The solution is evaporated to dryness and the residue fused.

H. F. E. H.

Testing Saccharimeters by Means of the Telescope Control Tube. C. A. Browne. (*J. Ind. and Eng. Chem.*, 1920, 12, 792-796.)—The telescope control tube consists of a metal tube which can be adjusted, by means of a screw, to give a column of solution of any length between 225 and 410 mm., the exact length at any point being indicated within 0.1 mm. by a vernier on a scale. In using the instrument to determine the errors of saccharimeter scales, due to inaccurate marking or to optical impurities in the quartz wedges, the scale of the tube itself is first calibrated, and a series of readings is made to prove that the zero point of the saccharimeter is perfectly adjusted. The control tube is then filled with a clear solution of about 25.5 grms. of sugar per 200 c.c. and placed in the trough of the saccharimeter. The temperature is maintained at about 20° C., and the polarisation closet ventilated by means of an electric fan to remove heat produced by the body of the observer. Five readings are taken successively for each scale division from the 100° point to the 95° point, and so on for every 5° to 55° point, and then back again to the 100° point. The readings are repeated with a fresh solution, and the average length of the tube for each scale division is corrected for errors in the tube scale. The corrected length, multiplied by 100 and divided by the corrected tube length for the 100° point of the saccharimeter, gives the value for that particular scale division. If, for example, the value 89.9 has been found for the 90° point, a deduction of 0.1° must be made for all readings near that point, and so on. The maximum error thus found in the

scale readings of three instruments by different makers was within the 0.05° limit fixed by the American Sugar Chemists, and the scales of commercial instruments should show this degree of accuracy. Other applications of the control tube are in comparing the scales of different saccharimeters, determining the probable error of observations of the observer, and estimating the influence of the personal equation. The general conclusion drawn from a long series of tests is that the quartz wedge saccharimeter, even in its most perfected form, has inherent defects which prevent its becoming an instrument of the highest precision.

C. A. M.



REVIEWS.

THE EXTRA PHARMACOPŒIA. By W. H. MARTINDALE, Ph.D., Ph.Ch., F.C.S., and W. W. WESTCOTT, M.B., D.P.H. Vol. I. Seventeenth Edition. 1920. H. K. Lewis and Co., Ltd., London, W.C. 1. Price 27s. net.

The appearance of a new edition of this well-known work, the first to be issued since the conclusion of the war, is very welcome. The book is necessarily of a highly technical character, but one cannot pass by the interesting Preface without wishing that a great part of it might be read by the man in the street, provided he had sufficient education to know what he was reading about. The authors would certainly not expect much appreciation from the average man, though they probably share the pious hope of the reviewer that ignorance of scientific facts may one day be a less usual attribute of Englishmen (see p. vi).

The Introduction contains useful tables and serves its purpose well; it is with the next 769 pages, wherein drugs and their uses are described, that the chief interest of the work lies. The reviewer, being a chemist, must necessarily deal principally with the chemical aspects of the book. The medical man will find data as to uses, dose, and methods of administration of the various materials, as well as remarks as to their incompatibility.

The chemist who takes an interest in materia medica, whether he adopts an academic or manufacturing point of view, will find a vast amount of useful information. Not merely can one find purely scientific chemical and physical data, but also many hints as to the manufacture of synthetic drugs, as well as much information relating to patents and trade-marks.

The really heroic attempt of the authors to provide "working formulæ for preparing various Organic Chemical Compounds" (Preface, pp. xvi-xviii) deserves the chemist's thanks; and the laboratory work involved in checking the "examples" of patent specifications must have entailed an enormous amount of labour. The reviewer has been able to check the advice given with regard to one or two preparations by his own results; in these cases he can fully endorse the remarks of the authors.

The treatment of certain compounds which have acquired prominence of late years calls for special mention.

Under "Acidum Hypochlorosum" we find not merely the uses of the acid itself and its salts described, but also good accounts of Chloramine—"T" and Di-Chloramine—"T"; it is hardly necessary to add that many references are given to the work of Carrel, Dakin, Watson-Cheyne, and other investigators, these antiseptics being treated in a manner which the reviewer, albeit only a chemist, found extremely interesting.

The heading "Acidum Salicylicum" covers the acid, its salts and derivatives, as well as the nearly related cresotinic acid; the 17 pages contain many references to patents and trade-marks.

"Organic Arsenic Compounds" take up 30 pages; it may be noted that the authors restrict the term "arsinic acid" to compounds of the type $R_3AsO(OH)$, whilst they refer to substances of the general formula $RAsO(OH)_2$ as "arsonic acids." On p. 204 we read:

"*Substitutes for Salvarsan* may be chemically identical, but clinically they differ remarkably." What is meant by the term "chemically identical" under such circumstances?

Twenty-five pages are devoted to "Coal-Tar Derivatives." This classification is logically weak, for here we find acriflavine, methylene-blue, phenacetin, phenazone, etc. (which are certainly derived from coal-tar), grouped together, whilst salicylic acid, equally a coal-tar derivative, has its own heading.

The account of "Colloidal Metals" occupies 21 pages; it should be pointed out that under this head colloidal solutions of various non-metals—*e.g.*, arsenic, iodine, selenium—are also described.

Other articles of considerable length are those devoted to "Hydrargyrum" (26 pages) and "Quinina" (17 pages).

A "Supplementary List of Drugs" occupies rather more than 60 pages; the reviewer feels that certain of these substances would have been better described in the main portion of the work.

The succeeding portions of the book are devoted to Vaccines and Antitoxins, Organotherapy, Poisons, and a Therapeutic Index of Diseases. As the book itself has two authors, one chemical and the other medical, so, for a complete review, it should have at least two reviewers; for an unaided specialised chemist realises his inability to deal with medical questions.

The Index, which has been tested in various ways, appears to be very complete. The printing is clear, an important matter where the use of so much small type is necessitated.

The authors are to be heartily congratulated on the appearance of this new edition.

J. T. HEWITT.

THE ANALYSIS OF DYESTUFFS AND THEIR IDENTIFICATION IN DYED AND COLOURED MATERIALS, LAKE-PIGMENTS, FOODSTUFFS, ETC. By ARTHUR G. GREEN, M.Sc., F.R.S., F.I.C. Third edition, 1920. London: Charles Griffin and Company, Ltd. Price 10s. 6d.

The first proposal for a scheme of analysis of dyestuffs was made by Witt in 1886, and this was followed by a more systematic method published by Weingärtner in 1887. In 1893 the author of this book advanced a more comprehensive scheme based on Weingärtner's system, and in the interval he has patiently carried out painstaking researches, which have culminated in a thoroughly scientific and practical programme for the detection of dyes of all classes both in substance and on the animal and vegetable fibre.

The book opens with a short but very lucid description of the hydrocarbons from which dyestuffs are derived, followed by mention of some of the more important intermediate products and an explanation of the conversion of certain of these into colouring matters. The relation between colour and chemical constitution and the meaning of "leuco" compounds are then discussed. All this is contained in 8 pages, but is by no means too short; it is exactly what the analyst or dyer requires as an introduction to the subject, and its clarity would have been obscured had its length been extended.

The classification of dyestuffs is then dealt with, both chemically and from the dyeing point of view, leading up very logically to an account of the analysis of colouring matters in substance. When it is remembered that in 1914 there were more than 200 blue dyestuffs on the market, it will readily be recognised that Professor Green is quite justified in saying that "All that can be reasonably expected of a scheme of analysis is that it should render a correct account of the dyeing and chemical relationships of the dyestuffs, and indicate in what direction the equivalent or identical products may be sought." True as this may be, yet it may be safely stated that the systematic and very practical methods of detecting dyestuffs which are expounded in this chapter should result in the identification of all but exceptionally obscure colouring matters. The important question of the detection of mixtures of dyestuffs is also adequately discussed. The paragraph on the detection of artificial dyestuffs in articles of food is of special interest to the analytical chemist. The detection of such colouring matters is described, but it appears to the reviewer that in a book entitled "Analysis of Dyestuffs" it would be very useful to find in this section exact details as to the analysis (estimation of lead, arsenic, etc.) of a selected number (say those permitted in the United States) of dyestuffs used for colouring foods.

Systematic tables, epitomising and exemplifying the principles laid down in this chapter and in the succeeding ones on the identification of dyestuffs on animal and vegetable fibres, form an important adjunct to the text.

A special chapter is devoted to the analysis of indigo, and its estimation, when dyed in conjunction with other colouring matters, is very completely described.

Now that organic dyestuffs, in the form of lakes, are so largely used instead of mineral pigments, these also have been brought into the scope of the book, accompanied by a tabular scheme of analysis.

The last chapter, a very interesting one, describes the methods employed for breaking up a dyestuff into simpler constituents in order to afford means of elucidating its constitution. Laboratory processes are given for reducing a number of azo-dyestuffs with stannous chloride or sodium hydrosulphite and identifying the products, and most of the bases and amino-acids which are commonly obtained on reduction are particularly described.

In this third edition the author has added a useful "Key to Trade Designations," based on a table prepared by Matthews (*Color Trade Journal*), which contains more than 200 trade names of groups of dyes, with their "dyeing class" and manufacturer's name; thus "Caledon" indicates vat dyes made by Scottish Dyes, Ltd.

The number of these trade names has been greatly increased of late, and the author, whilst excluding the more recent ones from the text and tables from consideration of space, has inserted the most important of them in the Index, with cross references to the products of German origin to which they are equivalent. The reviewer is inclined to agree with Professor Green in his opinion that many of these trade names are probably ephemeral, and will eventually give place to the name under which the products were known originally. Indanthrene blue, for example, has received no fewer than three new names since 1914—viz., Chloranthrene blue, Duranthrene blue, and Caledon blue—but there seems no reason, patriotic or otherwise, why the original name should not be maintained. Chemistry would indeed be reduced to chaos were this procedure applied to organic compounds to which an empirical name had first been given in Germany. It is to be hoped that dye manufacturers may be brought to agree that only one name should be used for each dyestuff—a simple name containing an indication of the “dyeing class” and the colour, with, perhaps, a distinguishing letter and number.

In this admirable monograph, which is quite a necessity for colour chemists, dyers, and analysts, there are singularly few misprints (p. 74, “Kupenfarbstoffe” instead of Küpenfarbstoffe; p. 107, “D. R. Staëble” instead of R. Staëble; p. 110, in the formula of dinitro-*a*-naphtholsulphonic acid only one nitro-group is indicated; p. 123, “Amido-Bronner” instead of Amido-Brönnner; and p. 133, “Alakli” instead of Alkali), but even after all these years the firm founded by Mr. Read Holliday of the curious Christian name is still, at the end of the Preface to the first edition, referred to as Messrs. Read, Holliday and Sons!

J. C. CAIN.

A TEXTBOOK OF QUANTITATIVE CHEMICAL ANALYSIS. By CUMMINS and KAY. 1919, Gurney and Jackson, London. Price 12s. 6d. net.

The fact that a third edition of this textbook, published originally in 1913, has been called for would indicate that it has met with general approval from students. In many respects the book is admirable, but we cannot help feeling that it fails to reach the ideal requisite for the systematic training of students in the fundamental principles of analytical chemistry. Whilst the explanations of the principles of analysis—the description of manipulative details, etc.—are clear and well put out, and cover quite elementary matter, the arrangement of the different sections of the book is confusing, and give one the impression that the authors had in a measure forgotten the real object and scope of the work. While admitting the educational value of the study of volumetric methods of analysis, we do not think that sufficient attention has been given to the systematic study of gravimetric work. The book would be much more valuable if the section dealing with gravimetric analysis was more fully dealt with, and that on systematic quantitative analysis completely rearranged. This section is arranged on alphabetical lines—a most undesirable plan; it surely would be more instructive to the student to have the elements arranged with some relation to their chemical affinities. In the section dealing with simple ores and alloys the methods described are sound and well thought out, but it is in

this section that the authors, when dealing with special technical matters, give unreliable information. As an example, under "Analysis of Superphosphate Manure," the authors make the statement that basic slag is one of the raw materials used in the manufacture of superphosphate, and apparently class all compound manures as "superphosphate." In describing the process for the determination of soluble phosphate in manures, the authors have not given correctly the official method of the Fertilisers and Feeding Stuffs Act.

Thirty pages of the text are devoted to water analysis, the excuse given for this being that students may find the determination of the small quantities of constituents normally present in water a useful exercise. We consider this a very dangerous idea—indeed, the tendency of the book is too much in the direction of training the student to specialise, and this to the sacrifice of the general principles of analytical chemistry. Although great advances have been made in the training of students at our universities and colleges, there is still much to be desired in this direction, and until analytical chemistry is fully recognised as an essential part of the study of chemistry, and not merely as a "side show," the training of chemists will be incomplete.

In more than one instance we note that in titrations the use of acid and alkali solutions of "normal" strength is indicated where very much more dilute solutions should be employed. The student is directed to read his burettes to 0.01 c.c., and to record weights to 0.1 of a milligramme. While we agree that it is very necessary to train the student to rigid accuracy in measuring and weighing, it is not desirable to lay stress on figures in the fourth place of decimals, which rarely have any significance.

E. W. VOELCKER.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

NOTES ON RUBBER ANALYSIS.

By A. R. PEARSON, B.Sc., F.I.C., LL.B.

THE following notes may be of use to chemists interested in the chemical testing of manufactured rubber goods :

1. *Determination of Total Sulphur*.—Numerous methods and modifications of methods have been used for determining total sulphur in rubber, and the very multiplicity of suggestions shows that the ideal is still to seek. An elaborate review of these methods is given by Tuttle and Isaacs in the *Journal of Industrial and Engineering Chemistry* (1915, VII., 658), and it is not proposed to criticise them in detail here.

The following modification of the nitric acid method has, after some years' experience, proved accurate and easy to carry out, and it requires no special apparatus, although it must be admitted that it leaves something to be desired as regards rapidity.

Twenty c.c. of fuming nitric acid (sp. gr. 1.5) are placed in a wide-necked flask of about 150 c.c. capacity, and exactly half a gram of the sample is dropped into the acid bit by bit, in pieces small enough to dissolve without fuming or inflaming. The flask is now closed with a small funnel and warmed by slow degrees, so as to avoid violent action and frothing. Finally, it is made quite hot on a water-bath or hot plate, and kept so for half-an-hour. At this stage the sulphur has probably all been oxidised to sulphuric acid, but it cannot be directly precipitated owing to the presence of complex nitro-compounds, which make filtration impossible. It remains, therefore, to get rid of the organic matter, and this can be conveniently done by means of potassium permanganate. The solid permanganate is added in small portions so as not to have the reaction too violent, and until some oxide of manganese remains unreduced after about an hour's heating. At the end of that time 20 c.c. of concentrated hydrochloric acid are added, and the liquid is again heated. When action has ceased, the solution is evaporated to dryness in a porcelain dish ; the residue is again treated with hydrochloric acid, and taken to dryness. The

mixed salts are now taken up in hot dilute hydrochloric acid, the solution is filtered and sulphates in it determined as barium sulphate.

The precipitated barium sulphate sometimes carries down a little manganese, but this can be removed by boiling the precipitate for a short time with dilute hydrochloric acid before washing.

All the reagents required for these operations are to be obtained practically free from sulphates; but it is advisable to carry out a blank experiment with every batch of estimations.

It is to be remarked that imperfect sampling is responsible for many errors and discrepancies in rubber analysis, and especially so in the determination of sulphur; for it is well known that sulphur has a tendency to accumulate near the surface of a vulcanised article. For example, in one case raspings from the surface of a sample whose average sulphur content was 2.5 per cent., yielded 5.4 per cent. of sulphur. Consequently the portion taken for a representative sample must consist of the whole of a section or sections right through the sheet or other article under examination. Rasping on a coarse carborundum wheel is a rapid and effective way of obtaining such a sample in a form suitable for analysis. Provided the sample is quite uniform and representative, duplicate determinations of sulphur by the above method never differ by more than 0.05 per cent.

2. *Determination of Carbonates in Rubber Mixings.*—Chalk and magnesium carbonate are frequently used as fillers in rubber manufacture, and it is therefore useful to know the percentage of carbonic acid in such a mixing.

An examination of the ash is useless for this purpose. The usual method is to dissolve away the rubber hydrocarbon in solvents such as nitrobenzene and examine the residue; but this is a tedious and not very reliable process. It is clearly preferable to decompose any such carbonates *in situ* by treating the rubber with some suitable acid. The aqueous mineral acids do not penetrate rubber, but acetic acid is sufficiently miscible with rubber to penetrate it completely either before or after vulcanisation, and so to decompose any carbonates which may be present. The carbon dioxide may then be purified, absorbed in soda lime, and weighed.

A convenient form of reaction-flask for this process is shown in the figure. Its capacity is about 100 c.c. A tap-funnel is ground into the neck, and a water-cooled reflux condenser is sealed to the side-tube. The absorption train which is connected at O consists of:

(1) A U-tube filled with solid neutral lead acetate, to absorb sulphuretted hydrogen.

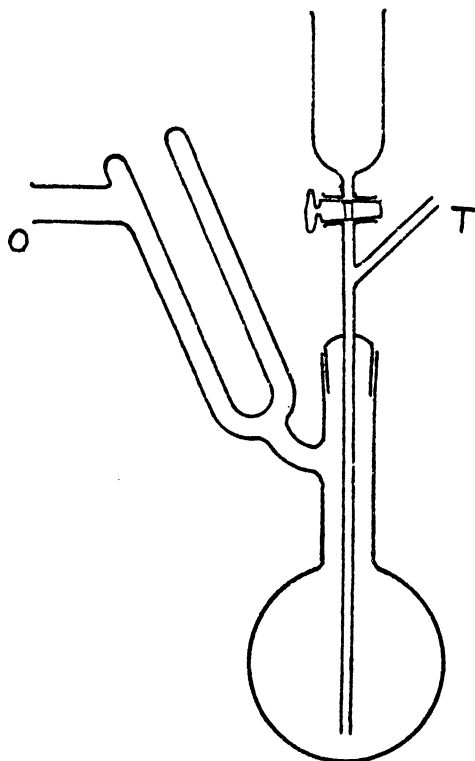
(2) A U-tube of which the first half contains sodium acetate which has been previously fused and powdered, and the second half contains calcium chloride. This arrests acid vapours and dries the gas. The purifying materials must of course be saturated with carbon dioxide before use.

(3) Two weighed tubes containing soda-lime and calcium chloride, to absorb the carbon dioxide.

One gram of the comminuted sample is introduced into the flask, and glacial acetic acid (25 c.c.) is run in by the tap-funnel. The mixture is heated so that it barely simmers, while a slow current of air, freed from carbon dioxide, is drawn

through the apparatus by way of the inlet tube T. The length of time required depends on the state of division of the sample. With raspings of the ordinary degree of fineness, and with a current of air passing at the rate of two bubbles per second, a treatment of one and a half hours is sufficient. The foot of the inlet tube should be protected by a small sheath-tube, otherwise it may become blocked by matter deposited from solution.

In calculating results it should be remembered that commercial magnesium car-



bonate is a basic carbonate of rather variable composition. The literature contains a number of suggested formulæ for it, and it generally has the approximate composition :

Magnesium oxide	...	...	42 per cent.
Carbon dioxide	...	...	34-35 "
Water of hydration	...	...	22-25 "

Since it is seldom possible to examine a sample of the carbonate used in a given mixing, some average factor must be employed to calculate, from the percentage of carbon dioxide found, the corresponding percentages of basic carbonate and of magnesium oxide: these two empirical factors are suggested :

$$\begin{aligned} \text{CO}_2 \times 2.8 &= \text{Basic carbonate.} \\ \text{CO}_2 \times 1.22 &= \text{Magnesia present in the form of carbonate.} \end{aligned}$$

3. *Determination of Sulphides in Rubber Mixings.*—The sulphides chiefly used as fillers are those of zinc and antimony. Lead sulphide also is sometimes found, for, although it is not often added as such, it is always formed to a greater or less extent during the vulcanisation of mixings containing litharge.

The coefficient of vulcanisation is a most important figure in rubber analysis, and as the sulphur of vulcanisation is usually determined by difference, it is necessary to know how much of the total sulphur is present in the form of sulphides.

As in the case of carbonates, a reagent is required which will penetrate the rubber and decompose these sulphides. Stevens (ANALYST, 1915, 40, 275) uses hydrochloric acid in a medium of benzene or ether, and determines iodimetrically the sulphuretted hydrogen which is evolved. Another method which has been found useful, where a considerable number of analyses is to be done, consists in decomposing the sulphides with a mixture of acetic and hydrochloric acids and absorbing the sulphuretted hydrogen by solid copper sulphate, so that it can be weighed directly.

The reaction is carried out in a flask similar to that described above. After the sample has been introduced, nitrogen from a cylinder fitted with reducing valve is passed through the flask until all air is displaced. Then the absorption train is connected, and a mixture of acetic acid (15 c.c.), concentrated hydrochloric acid (5 c.c.), and water (5 c.c.), is run in by the tap-funnel. The absorption train consists of :

1. A tube packed with sodium acetate and calcium chloride as described above. This should be saturated with sulphuretted hydrogen, and then filled with nitrogen before use. The same tube should not be used for determination of carbonates and of sulphides.

2. A weighed U-tube for absorbing the sulphuretted hydrogen. This is filled, as to its first two-thirds, with copper sulphate in fine crystals which has been dried at 105° C. ($\text{CuSO}_4 \cdot \text{H}_2\text{O}$), and then, as to its last third, with calcium chloride. The air in this tube must be displaced by nitrogen before weighing.

The operation is carried out in the same way as the determination of carbonates above. The following points are worthy of remark :

1. Air must be excluded as far as possible, because the copper sulphide absorbs oxygen slowly. It is inevitable that a little air should remain in the connections of the apparatus, and even this small amount causes results to become slightly higher when the sulphide accumulates from repeated experiments. This is shown in the following series of test experiments, in which a mixture of zinc sulphide and zinc oxide was used :

Experiment.	Weight of Mixture.	Weight of H_2S found.	Percentage of H_2S .	Percentage of ZnS .
1	0.4866 grm.	0.0784 grm.	16.11	46.07
2	0.2392 "	0.0386 "	16.14	46.13
3	0.4022 "	0.0654 "	16.26	46.49
Actual percentages:		...	16.09	46.00

It is advisable, therefore, to carry out a blank experiment from time to time, and to make a corresponding deduction if any increase in weight is observed.

2. A nitrogen atmosphere is preferable to one of carbon dioxide, because the sodium acetate and calcium chloride always contain traces of basic compounds, and variable equilibria might be set up, such as : $\text{Na}_2\text{CO}_3 + \text{H}_2\text{S} \rightleftharpoons \text{Na}_2\text{S} + \text{H}_2\text{O} + \text{CO}_2$.

3. None of the combined sulphur is expelled by this treatment, for soft vulcanised rubber containing practically no mineral matter yields no sulphuretted hydrogen.

4. Commercial zinc sulphide may contain notable quantities of zinc oxide, free sulphur, and other substances. Commercial antimony sulphide is also of variable composition: it may contain trisulphide, pentasulphide, and basic compounds of antimony, together with free sulphur and compounds of lime and alkalies. Of course, only that sulphur corresponding to the trisulphide is estimated by the above method; but it is precisely this sulphur which is not available for the reaction of vulcanisation.

240, GOSWELL ROAD, E.C.



AN IMPROVEMENT IN THE HALDANE GENERAL AIR ANALYSIS APPARATUS.

By ROBERT C. FREDERICK.

For general air analysis the writer considers the Haldane apparatus to have no superior, and in an extensive experience of its use a number of minor improvements in arrangement and manipulation have suggested themselves. Some of these have been referred to elsewhere,* and this note concerns a small modification which, gives distinct practical advantages.

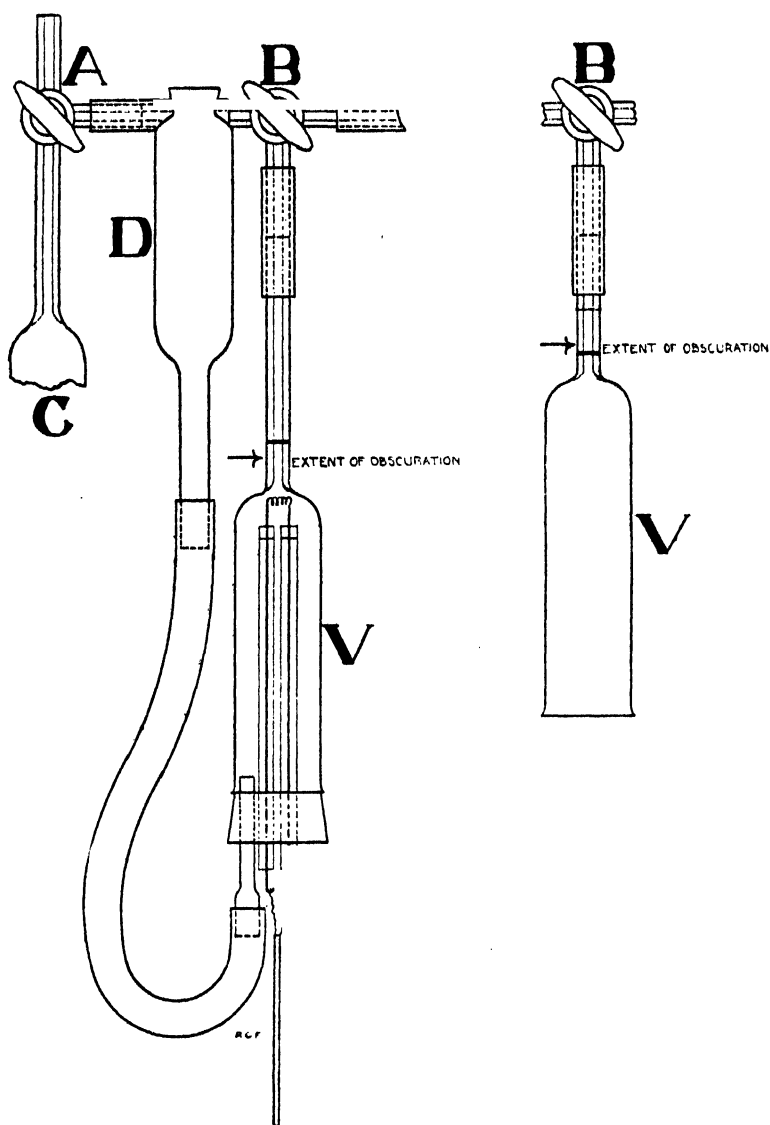
In brief, this improvement consists of using a combustion chamber V in which the wide part is $\frac{3}{8}$ inch shorter in length, the stem $1\frac{1}{8}$ inches longer, and the mark on the stem is $\frac{1}{2}$ inch from the shoulder ($\frac{3}{8}$ inch higher); the fitting of this does not necessitate any rearrangement of the other parts.

The object is twofold. In practice it is found that the lower part of the stem of the combustion chamber rapidly becomes obscured by the combustions, and in the original this obscuration extends over the graduation, so that after very little use the part has to be dismantled and cleaned; with V modified, as shown, the obscuration remains well below the graduation, and this frequent cleaning is not required.

Again, in using this apparatus for examination of samples contained in bottles, these are opened under mercury, and the air is transferred by means of a bent tube with a three-way stopcock. In this manipulation a globule of mercury is almost invariably retained in the stopcock A of the apparatus and must be removed. The writer does this by turning A open from C (air burette) to B only, and B open from V towards A only, and then, by gently pressing the two limbs of the rubber tube connecting D and V against each, displacing the globule. In doing this with the

* "Public Health Chemical Analysis" (Constable and Co.).

original apparatus it frequently happens that the mercury is pushed up in the stem of V until it reaches the stopcock B, without freeing the globule from the narrow



Scale, one half.

tube below A ; the increased length of the stem of V enables the displacement to be made without difficulty.

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NOTES.

The Editor desires to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

SOUR MILK SAMPLES AND GERBER'S FAT METHOD.

I HAVE recently had occasion to re-test, after a lapse of several weeks, a number of milk samples for fat by Gerber's method. These had been "preserved" by the addition of about 0.05 per cent. of formalin, which of course is not effectual for more than a few days, and the samples were very sour and decomposed. About 2 per cent. of strong ammonia was added to make pipetting possible, and, after correction for this, the results were up to 0.2 per cent. higher than those of the original tests. As would be expected, the ammonia is not responsible for this, as eight tests on five different milks averaged—

With ammonia	...	...	3.67
With ammonia, corrected	...	...	3.74
Without ammonia	...	...	3.76

The possibility of the formation of amyl esters suggested itself, and a sample of milk was divided into several parts, to which acetic, lactic, and butyric acids in known amounts were added. To make all comparable, 0.05 per cent. of formalin was also added, and 2 per cent. strong ammonia to neutralise the acidity. The following table shows the apparent increase in fat over the control, also the apparent increase in the case of several sour samples tested at the same time.

						Total Acid (as Lactic Acid).	Approximate Increase in Fat per Cent.
Milk +	0.5	per cent.	acetic acid	...	...	0.57	0.10
" +	0.5	"	butyric	"	...	0.42	0.32
" +	1.0	"	"	"	...	0.63	0.57
" +	0.5	"	lactic	"	...	0.70	0.05
" +	1.0	"	"	"	...	1.20	Nil
" +	0.17	"	acetic	"	}	0.57	0.12
	0.17	"	butyric	"			
	0.17	"	lactic	"			
" +	0.17	"	acetic	"	}	0.80	0.19
	0.33	"	butyric	"			
	0.33	"	lactic	"			
Milk	3 days old,	no smell of butyric acid				0.72	- 0.06
"	1 week old,	" " "				1.02	0.18
"	3 weeks old,	smell of butyric acid				1.12	0.12
"	5	"	"	"	"	2.10	0.19

The results in the case of the artificially soured samples were corrected for the volume of acid added, and those of the naturally sour samples for the ammonia. The fat separated from the most acid samples had a distinct odour of amyl esters.

It is evident that the use of Gerber's method for sour milk is apt to give very

misleading results, owing to the formation of amyl esters of the lower aliphatic acids. Lactic acid appears to have no effect, acetic acid little, but butyric acid a large effect. I was unable to try the effect of propionic acid, but one would expect it to be intermediate between that of acetic and butyric acids, in which case it would be an important source of error.

F. E. DAY.

LANSDOWNE, LIMERICK.

THE IDENTIFICATION OF ACIDS BY PHENACYL BROMIDE.

A useful method for the identification of organic acids by means of the m-pt. of their phenacyl esters has been described by Rather and Reid (*J. Amer. Chem. Soc.*, 1919, 41, 75) and abstracted in the *ANALYST* (1919, 44, 103).

The method depends upon the use of ω -bromoacetophenone for the esterification of the acids; and the writer thinks it worth while to call attention to the fact—learned by unpleasant experience—that special care should be taken in preparing and using this substance and its solution. Even small quantities produce very painful blisters on the skin if allowed to come in contact therewith. The substance also has a very irritating effect upon the eyes.

H. E. Cox.

NEWPORT, MON.

MASKING THE PRESENCE OF FREE SALICYLIC ACID IN ACETYL SALICYLIC ACID.

The writer, (*Pharm. J.*, 1920, 51, 90), in a paper describing a new method for the estimation of free acetic acid in acetyl salicylic acid, also drew attention to the masking of the free salicylic acid present in aspirin tablets when tested for with ferric chloride test solution. The substance causing this was tartaric acid, some colourless non-ionised salt of iron being formed, there being no free ferric ions available for the formation of the violet-coloured ferric salicylate. Certain other strong acids will produce the same phenomenon, particularly citric acid (1.0 per cent. of the latter being sufficient to mask the presence of about 0.2 per cent. of free salicylic acid).

Tartaric acid, citric acid, etc., when present, can readily be detected after removal of the salicylic acid, acetyl salicylic acid, etc., from a cold aqueous extract of the tablet, and, conversely, the free salicylic acid can be estimated colorimetrically, after separation by means of some suitable solvent, such as ether, chloroform, etc., which does not extract the masking substance; when either tartaric or citric acid is present, a mixture of equal parts of ether and petroleum ether is a better solvent than ether alone, since both acids are slightly soluble in ether. All samples of aspirin tablets, therefore, which, after being powdered, give no violet coloration with ferric chloride test solution, should be viewed with suspicion.

A. NUTTER SMITH.

MESSRS. BOOTS' PURE DRUG CO., LTD.
NOTTINGHAM.

THE TREATMENT OF RED LITMUS PAPER.

Owing to the sizing of the original paper with rosin (colophony), it is frequently found that red bibulous litmus papers are not readily wetted by aqueous solutions, and that the colour indication is indefinite and slow in appearance.

This trouble may be obviated by the following procedure: A number of litmus books are rolled into a double layer of filter paper and inserted, backs downwards, in a Soxhlet extractor. They are then covered with 85 per cent. (or stronger) alcohol,

containing 1 per cent. of concentrated hydrochloric acid, and allowed to soak overnight, when a further quantity of acid alcohol is added, and extraction carried through in the usual manner, until no more red colouring matter is removed. The books are then squeezed dry in a cloth, and finally dried in an oven at 100° C. The alcohol removes, in addition to the rosin, some of the colour from the litmus and some from the red dye of the bookbinding.

T. J. WARD.

THE LABORATORY,
STAG BREWERY, S.W. 1.



ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Detection and Identification of Dextrose in Vegetables by a Biochemical Method. E. Bourquelot and Bridel. (*J. Pharm. Chim.*, 1920, **22**, 209-215.)—The method depends on the formation of methyl glucoside when dextrose in methyl alcohol solution is treated with emulsin. For instance, a solution containing 2.0565 grms. of dextrose per 100 c.c. of 70 per cent. methyl alcohol (by weight) showed a rotation of +2° 20' in a 200 mm. tube; this solution was then treated with 0.5 gm. of emulsin, and placed aside at the ordinary temperature. The rotation decreased gradually (during some days) to a final value of -50', and 82.5 per cent. of the dextrose had combined with the alcohol. Experiments with mannose, lævulose, and arabinose showed that these sugars do not combine with methyl alcohol. The methyl glucoside may be separated by evaporating the alcoholic solution under reduced pressure, and extracting the residue with boiling ethyl acetate; crystals of the glucoside form as the solution cools. The method may be applied to the detection of dextrose in the reducing sugars isolated from plants and vegetables; the author finds that the reducing sugars from juniper berries contain 15 per cent. of their weight of dextrose.

W. P. S.

Estimation of Methyl Alcohol in Cacao Nibs and Husks. Von Koers. (*Pharm. Weekbl.*, 1920, **57**, 924; through *Chem. Zeit. Rep.*, 1920, **44**, 234.)—Von Fellenberg proposed to estimate the amount of adulteration in cacao by determining the methyl alcohol content after liberation by pectin. The author finds the same values as Von Fellenberg for nibs, but lower ones for husks and for certain cocoas on the market; hence the estimation of husk by this method appears impracticable.

O. E. M.

Hegner Value of Fats and Oils. Goldschmidt. (*Zeitsch. Öl-Fett-Ind.*, 1920, **40**, 406; through *Chem. Zeit. Rep.*, 1920, **44**, 244.)—The following procedure is recommended for the determination of this value. Five grms. of the sample are saponified, unsaponifiable matters are extracted from the soap solution in the usual way, and the solution is then acidified and the fatty acids extracted with ether. The ethereal solution is dried for two hours over 10 grms. of anhydrous sodium sulphate, filtered

into a weighed flask, and the sodium sulphate and filter are washed with ether (also dried over sodium sulphate). The greater part of the ether is removed by distillation, and the residue of fatty acids is dried at 50° to 60° C. in a current of air.

W. P. S.

Estimation of Oxy-Fatty Acids in Extracted Olive Oils. Stadlinger. (*Zeitsch. Öl-Fett-Ind.*, 1920, 40, 437; through *Chem. Zeit. Rep.*, 1920, 44, 244.)—A grade of oil obtained by extracting olive residues with carbon bisulphide is particularly rich in oxy-fatty acids, the average quantity of the latter being 6 per cent. To estimate the amount of oxy-fatty acids, the total fatty acids obtained from the oil are melted, treated with petroleum spirit (b.pt. below 50° C.); the insoluble oxy-fatty acids are collected on a filter, washed with petroleum spirit, dissolved in warm alcohol (or a mixture of alcohol and chloroform), the solution evaporated, and the residue of oxy-fatty acids weighed.

W. P. S.

Use of the Refractometer in Ascertaining the Purity of Certain Refined Edible Oils. F. H. Trim. (*J. Soc. Chem. Ind.*, 1920, 39, 307-308 T.)—The melting-point, determined under standard conditions, will afford an indication of the amounts of constituents in dual mixtures, such as palm-kernel oil or coconut oil with each other or with arachis oil. In the case of mixtures of all three fats another constant is also required, and for this purpose the refractive index is the most useful. Standard samples should be prepared from the original material by extraction or expression, and the crude oils so obtained treated by laboratory methods similar to those used in the works. The following results were thus obtained with refined edible oils:

Oil.	Standard [n] _D 40° C.	Variation.	Melting-Point ° C.	Free Fatty Acids (as Lauric), per Cent.
Arachis ...	1.46235	± 0.00020	- 8.0	0.05
Palm-kernel ...	1.45097	± 0.00020	28.35	0.04
Coconut ...	1.44897	± 0.00010	24.85	0.03

Coconut oil showed the least variation in refractive index. The following results were given by refined oils from four different copras: Ceylon, 1.44887; Straits, 1.44902; Singapore, 1.44887; and Malabar, 1.44897. The melting-points and refractive indices of dual mixtures of pure arachis, palm-kernel, and coconut oil were plotted in graphs against the percentage compositions. The results showed that the refractive index was directly proportional to the percentage composition, and that the addition of 10 per cent. of arachis oil lowered the melting-point by only 0.4° C. If the refractive indices are plotted as ordinates and the melting-points as abscissæ a closed graph is obtained. This may be divided into sections, and any particular section used for the estimation of the constituents of either dual or triple mixtures. Allowances must be made, however, for natural variations in the natural products having an influence on the constants of the refined material.

C. A. M.

New Method of Estimating Quinine. C. Bamberger. (*Pharm. Zentralb.*, 1920, 61, 257-259; through *Chem. Zeit.*, 1920, 44, *Rep.*, 223.)—Two and a half grms. of cinchona bark are heated for ten minutes over the water-bath with 2 c.c. of hydrochloric acid and 20 c.c. of water, and the mixture then cooled, shaken with 25 grms. of chloroform and 50 grms. of ether, and treated with 5 grms. of sodium hydroxide solution. The contents of the flask are vigorously shaken (about 300 times) within two to three minutes, and treated with sufficient plaster of Paris (about 40 to 50 grms.) to clarify the liquid, and 60 grms. of the clear chloroform-ether layer transferred to a separating funnel. It is shaken twice (for two minutes each time) with 5 c.c. of $\frac{N}{10}$ hydrochloric acid, and twice with 10 c.c. of water, and the extracts united and titrated with $\frac{N}{10}$ potassium hydroxide solution, methyl red being used as indicator. The results agree closely with those obtained by the Swiss Pharmacopœia method, and the methods of Frerichs and Mannheim. C. A. M.

Laboratory Control of Wheat Flour Milling. B. R. Jacobs and O. S. Rask. (*J. Ind. and Eng. Chem.*, 1920, 12, 899-903.)—The authors discuss fully the composition of the wheat kernel and its component parts, and describe methods employed in mill control and the examination of commercial mill products. Methods for determining the "flour content" of wheat and flour mill products are fully entered into, while calculations are embodied which may be employed to check miller's yield reports. It is shown that the amount of fat and pentosans in any grade of flour is a measure of the amount of offal (germ and bran) contained in that flour. The amount of starch contained in wheat or any mill product is a measure of the amount of inner endosperm, and therefore of the amount of flour contained in that product while the determinations of pentosans and starch may be used as a control of the miller's yield reports, various formulæ being given which are suitable for such a calculation. H. F. E. H.

Analysis of Wines. W. Fresenius and L. Grünhut. (*Zeitsch. anal. Chem.*, 1920, 59, 209-232.)—Directions are given for the estimation of ash, total alkalinity of the ash, alkalinity of the soluble ash, and phosphoric acid (see also ANALYST 1920, 304). W. P. S.

Detection and Estimation of Yellow AB and Yellow OB in Mixtures. W. E. Mathewson. (*J. Ind. and Eng. Chem.* 1920, 12, 883.)—These dyes, benzeneazo- β -naphthylamine (AB) and o-tolueneazo- β -naphthylamine (OB) are the only oil-soluble coal-tar colours allowed in food products by the U.S. Department of Agriculture. The usual liquid mixtures sold for colouring purposes consist of 2 to 3 per cent. solutions of the dyes in oil, and are tested by diluting with from 500 to 1,000 times their volume of petroleum spirit, extracting 30 c.c. with 1 or 2 c.c. of 7N sulphuric acid, followed by two extractions with 18N acid, and finally, if the petroleum spirit still contains colour, with 24N acid. The 18N extract contains practically all the yellow AB and the yellow OB. Carrotin and annatto remain in the petroleum spirit after shaking with 24N acid, although bixin is removed by acid of this strength. The 18N acid extracts after dilution are submitted to an elaborate

series of extractions and distillation, and the dyes are finally obtained in 100 c.c. of 93 per cent. alcohol. The light absorption constant at wave length 0.436μ is then determined by means of a spectrophotometer, using a quartz mercury lamp as the source of illumination. By careful measurement it is possible to estimate the proportion of each dye present. Numerous details of experimental work performed in the working out of the above method are described, and a few colour tests are given, which, however, are of little value when mixtures of the two dyes are present, owing to similar results being given by each.

T. J. W.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Determination of Cellulose in Woods. S. A. Mahood. (*J. Ind. and Eng. Chem.*, 1920, 12, 873-875.)—A uniform size of particle is essential, if comparable results are to be obtained in the determination of cellulose in woods. Material which passes an 80-mesh standard (American Society of Civil Engineers) sieve, but is retained on a 100-mesh sieve, has been found to be most satisfactory from the standpoint of both yield and manipulation. Material obtained by a single mechanical process of disintegration may give a sample on sifting which is not representative; to avoid this, a combination of two processes, sawing and grinding, has been used. The procedure recommended by Sieber and Walter—viz., allowing the material to remain in the crucible throughout the treatment with chlorine and sodium sulphite for chlorination—gives a lower yield of cellulose than the original Cross and Bevan method, probably because of the higher temperature at which chlorination takes place. The modification of the Cross and Bevan method proposed by Johnsen and Hovey, who subject the wood samples to a preliminary hydrolysis with acetic acid in glycerol at $135-140^{\circ}\text{C}$., appears to be of doubtful value, since the cellulose as well as the hemi-celluloses and furfural-yielding constituents are attacked. Dore, in a recent article (*J. Ind. and Chem. Eng.*, 1920, 12, 264), shows that the more resistant portion of the cellulose, the α -cellulose, is attacked by the acetic acid-glycerol mixture. Experiments are described giving the results obtained for cellulose with varying the size of the material between 40 and 120 mesh, and also comparative determinations when using the methods of Johnsen and Hovey (Sieber and Walter's apparatus), and of Cross and Bevan as modified by Renker both in Schorger's and in Sieber and Walter's apparatus.

H. F. E. H.

Determination of Phytin in Plant Extracts. A. Rippel. (*Biochem. Zeitsch.*, 1920, 103, 163-172; through *J. Soc. Chem. Ind.*, 1920, 39, 582A.)—Phytin can be precipitated more or less quantitatively in the presence of inorganic phosphates by copper acetate in acetic acid solution. Satisfactory results can only be obtained with definite concentrations of copper acetate and acetic acid. An extract from barley yielded 63.3 per cent. of the phytin P_2O_5 by precipitation with copper acetate in 1 per cent. acetic acid. The same method accounted for 92.8 per cent. of the phytin in standard solutions of that substance. The phytin in an acetic acid extract from peas could not be precipitated by copper acetate at all.

ORGANIC ANALYSIS.

Rapid Method of Estimating Aromatic Hydrocarbons in Petroleum Spirits. W. Hess. (*Zeitsch. angew. Chem.*, 1920, **33**, 147, 176; through *Chem. Zeit.*, 1920, **44**, *Rep.*, 259.)—A method of estimating aromatic hydrocarbons in petroleum spirit is based on their conversion into mono-nitro-compounds, and their separation from the unchanged non-nitrated hydrocarbons by means of concentrated sulphuric acid, in which the nitrated hydrocarbons are soluble. An apparatus of special form is used for the estimation, in which it is essential that the relative proportions of the reacting substances and the concentration of the acid should be kept as constant as possible. The nitric acid used is freed from nitrous acid by means of a current of air at 40° to 45° C. If necessary, a temperature correction must be applied when reading the volume of non-nitrated hydrocarbons. C. A. M.

Estimation of Benzine in Turpentine. A. Allina and H. Salvatena. (*Chem.-Zeit.*, 1920, **44**, 673-674 and 697-698.)—The methods of Herzfeld and Marcusson depending on the destruction of the terpenes are subject to large errors. The authors have investigated four methods:

(1) Temperature rise with sulphuric acid. The sample, 100 c.c., is stirred with a thermometer in a smaller beaker, enclosed with glass wool packing in a larger one, with 10 c.c. of 2:1 sulphuric acid. The rises of temperature, of which a table is given, varied from 2° C. for substitute to 137° C. for fresh Neustadt turpentine.

(2) Specific dilatation. Although the terpenes have a lower coefficient than benzine the method has no quantitative value.

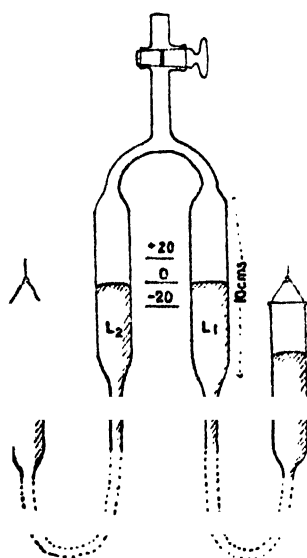
(3) Raising the sp. gr. by bromination. Irregularities in bromination, which was carried out by shaking with potassium bromide and bromate, with addition of hydrochloric acid, confine the method to distinguishing between genuine turpentine and substitute, giving an approximate value for the benzine.

(4) Bromination followed by steam distillation. The boiling-point of the terpenes is raised, whatever the nature of the bromination (substitution or addition), so that irregularities in this respect do not invalidate the method.

The bromination is carried out in a Claisen flask with 20 grms. of potassium bromate in 10 c.c. of water; 20 c.c. of the sample, and 50 c.c. hydrobromic acid (50 per cent.), 1 c.c. at a time, are added with shaking, and with cooling at first. Later, a reflux condenser being attached, the temperature is allowed to reach 40-50° C., at which it stands for an hour. The liquid is then decolorised with sodium carbonate and arsenic trioxide in powder. The side limb of the flask is filled with beads and glass wool for the steam distillation. A U-tube with one graduated limb (20 c.c. by tenths) surmounted by a cock and funnel, and having a cock below the graduations, a run-off cock at the lowest point, and another in the plain limb level with the highest graduation, is used as a receiver. This is filled with water, and the quantity of benzine per 5 c.c. of water coming over is noted. When distillate heavier than water, which may carry down benzine, appears, the water is run off, and after shaking with fuming sulphuric acid, the quantity of benzine is noted. Up

to 50 per cent. of benzine, 40 c.c. of condensed water suffices, but more is necessary for higher contents. The use of a water-cooled Hempel column for the distillation is recommended. A table is appended showing results, ranging from 11.1 to 89.4 per cent. respectively for 10 and 90 per cent. benzine taken in the experimental sample, with 40 c.c. condensed water.

O. E. M.



Consistency of Greases. F. S. Clulow and C. W. Taylor. (*J. Soc. Chem. Ind.*, 1920, 39, 291-295T.)—The viscosity or consistency of a thick grease at a definite temperature is reduced when the grease is used under working conditions, although the temperature remains constant. The apparatus shown in the illustration was employed to measure any change of viscosity due to mechanical treatment. The two mercury reservoirs, R_1 and R_2 , are connected by means of rubber pressure tubing to the lower ends of the tubes L_1 and L_2 , and the reservoirs are connected by a gut string passing over small pulleys, so that when one moves upwards the other moves downwards by an equal amount. The apparatus is surrounded by a water jacket. To use the apparatus, the temperature of the water jacket is raised a few degrees above the melting-point of the grease, both reservoirs are raised, with the tap of the apparatus open, so as to fill the tubes L_1 and L_2 with mercury, and the melted grease is poured in through

the tap, the reservoirs being lowered gradually until the mercury in both bulbs is at the zero mark. The tap is closed and the grease brought to the temperature at which the determination is to be made. The right-hand reservoir is then brought to the zero mark, and the gut string so adjusted that the mercury level in the left-hand reservoir is 140 mm. below this level. As the mercury level in the right limb L_1 moves upwards, the right reservoir is raised slowly, keeping the mercury surfaces level; the left reservoir descends at the same rate. The time noted is that required for the mercury surface to move 20 mm. above and below zero in either limb. By reversing the process the grease is forced in the opposite direction through the apparatus. The rate of flow of the grease increases each time it is forced through the tube, until a constant time is at last obtained which cannot be decreased by further working. For instance, at 32° C., a specimen of grease had an initial time of flow of 720 seconds, but after some fifty passages through the tube the time of flow was reduced to 15 seconds (constant). When re-melted and cooled to 32° C., the grease regained its high initial viscosity, which gradually dropped to the minimum value after working. This decrease of viscosity, brought about by mechanical treatment, is due to the destruction of the original crystalline structure of the grease. The apparatus may be standardised against castor oil, and it is suggested that such a head of mercury should be used as will produce a time value of 13.5 seconds for castor oil at 0° C.

W. P. S.

Reactions of Dichlorodiethyl Sulphide ("Mustard Gas"). F. Martin. (*J. Pharm. Chim.*, 1920, 22, 161-165.)—Dichlorodiethyl sulphide is oxidised by nitric acid and other oxidising substances, and the solution obtained yields the usual reactions for sulphuric acid. If a few drops of dichlorodiethyl sulphide are heated with potassium hydroxide solution, the vapours produced give a white precipitate with Dénigès' reagent (an acid solution of mercuric sulphate). When the vapours are absorbed in water an odour resembling that of mercaptan is observed on the addition of a drop of iodine solution. The substance is hydrolysed by water, especially by acidified water, and the solution absorbs bromine. The presence of a few cgrms. of the substance in 1 litre of water may be detected by means of a solution of 8 grms. of potassium iodide, 0.8 grm. of copper sulphate, and 1 c.c. of gum arabic solution in 33 c.c. of water; this reagent is set aside for two hours, and then filtered before use. On mixing the aqueous solution of dichlorodiethyl sulphide with one-fourth of its volume of the reagent, a white turbidity develops, due to the formation of diiododiethyl sulphide. If the gum arabic is omitted from the reagent the reaction is slower, and the diiododiethyl sulphide separates in the form of crystals. W. P. S.

Determination of the Jellying Power of Gelatines and Glues by the Polarimeter. C. R. Smith. (*J. Ind. and Eng. Chem.*, 1920, 12, 879.)—The optical rotation of solutions of all gelatines and glues at 35° C. is practically constant, ranging from -6.70 to -7.20 degrees Ventzke per grm. of solid material, but on cooling to 15° C. the rotation increases proportionately to the viscosity of the solution. Increase in concentration gives higher values, but by keeping the solutions for several hours at 15° C. a gradual decrease is observed, so that solutions of all strengths under the above working conditions yield a constant rotational value for any particular sample. Determinations of the polarimetric values are made by soaking 3 grms. of the powdered air-dried gelatine in cold water for thirty minutes, immersing in a boiling water-bath until solution is complete, cooling to 35° C. and diluting to 100 c.c. If clarification is necessary 5 grms. of light magnesium carbonate are added to the solution, which is kept at 30-40° C. for an hour, and filtered bright. The first rotation is measured in a 200 mm. tube at 35° C., the solution then being kept at 15° C. overnight, and the rotation again observed next day. The results are expressed by the formula $\frac{\text{Rotation at } 15^{\circ}}{\text{Rotation at } 35^{\circ}}$ and are inversely proportional to the percentage of gelatine required to produce a standard jelly at 15° C. This standard is defined as a solution of such strength that a bubble of air 4 to 5 mm. diameter admitted to the polarimeter tube moves vertically with a motion of 4 cm. per second. Two tables are provided, giving a large number of results obtained, and a third one showing the comparison between the polarimetric values and jelly strengths obtained in the following manner: A 60° funnel is partly filled with mercury, 50 c.c. of the gelatine solution are poured upon the surface, and allowed to set at 10° C. The mercury is then run out, and a partial vacuum of 600 mm. of water produced below the jelly, when the depression of the upper surface is measured by a micrometer gauge. The results obtained by this method bear a moderately definite ratio to those given by the polarimetric method. T. J. W.

Application of Devarda's Method to the Estimation of Nitrogen in Nitroglycerine. Koehler, Marqueyrol, and Jovinet. (*Ann. Chim. anal.*, 1920, 2, 271-272).—A quantity of about 0.5 gm. of nitroglycerine is mixed with 40 c.c. of hydrogen peroxide (12 vols.) and 5 c.c. of potassium hydroxide solution in a conical flask, and the mixture is heated at 40° C. for not less than forty minutes. The mixture is agitated thoroughly during this period by means of a mechanical stirrer (1,500 revs. per minute). The inside of the flask is then rinsed down with a few c.c. of alcohol, and the heating continued for a very short time at 70° C. The subsequent reduction of the nitrate with Devarda's alloy and the distillation of the resulting ammonia are carried out in the usual way. W. P. S.

Relation between the Quantities of Tarry and Carbonised Substances in Heavy Mineral Oils. W. Steinkopf and H. Winternitz. (*J. prakt. Chem.*, 1920, 101, 82-92).—A large number of experiments were made with the object of determining to what extent the formation of carbonised matter (coke) in heavy mineral oils is influenced by the amount of hard and soft tarry substances present, and also to determine the difference of coke formation at ordinary pressure and *in vacuo*. The hard tarry substance was estimated by Holde's method and the soft tarry substances by Holde and Meyerheim's method (*ANALYST*, 1911, 36, 298); the distillations for the estimation of carbonised matter were made in an atmosphere of nitrogen, and during the vacuum distillations the flask was shaken continuously. Generally, an oil containing a large quantity (2 to 6 per cent.) of hard tarry substances yields a considerable amount of carbonised matter (5 to 8 per cent.); but in the case of a few oils (Argentina), from 40 to 60 per cent. of the soft tarry matters are carbonised during distillation. Excepting very tarry oils, the results obtained by distillation at ordinary temperature and *in vacuo* differed to a slight extent only as regards the yields of carbonised matter. W. P. S.

Determination of the Specific Heat of Heavy Mineral Oils. H. S. Bailey and C. B. Edwards. (*J. Ind. and Eng. Chem.*, 1920, 12, 891-894).—The method depends upon the correct determination of the electrical energy required to heat a definite mass of oil through a given range of temperature. Determinations can be carried out rapidly at any temperature below the point where the vaporisation of the sample causes an appreciable error, the results being satisfactory for the calculation of heat transference data in oil circulating systems. The apparatus employed is described in detail, together with the methods of calculating results: 250 grms. of the oil are employed in the calorimeter, which is a 1-quart vacuum-jacketed glass vessel 25 ins. in diameter and 11½ ins. deep enclosed in a copper jacket, sold under the name of "Thermos Fruit Jar." Accuracy of any desired degree may be obtained by refinements in temperature and electrical energy measurements.

H. F. E. H.

Commercial Examination of Sulphonated Oils. G. F. Pickering. (*J. Soc. Chem. Ind.*, 1920, 39, 305-306r).—A few c.c. of the sample are boiled with three times the volume of hydrochloric acid until the fatty layer is clear, and the acid layer

is then drawn off and tested with barium chloride, a precipitate indicating the presence of sulphonated oil. If the oil layer is charred, the methods suitable for oils of high iodine value must be used. *Water*.—Distillation with xylene is the most accurate method, but in the case of oils of low iodine value it is sufficient to heat about 2 grms. in a $1\frac{1}{2}$ in. Petri dish in the water-oven. The amount of any ammonia present must be deducted from the loss of weight. *Total Fat in Oils of Low Iodine Value*.—About 5 grms. are boiled with dilute (1 : 1) hydrochloric acid until the fatty layer is clear, and, after cooling, extracted twice with ether. The united extracts are washed free from acid and distilled, and the residue of fat dried in the water-oven. *Oils of High Iodine Value*.—Five grms. are dissolved in 50 c.c. of pyridine, and the solution heated on the water-bath with 25 c.c. of strong hydrochloric acid for at least one and a half hours, with frequent shaking. It is then cooled, and extracted twice with ether, as described above. *Total Sulphuric Acid*.—About 2 grms. of the sample are distributed in small drops over a thin layer of powdered sodium carbonate in a platinum dish, and then covered with a layer of about $\frac{1}{4}$ in. of the carbonate, and then heated, first over a luminous Argand flame until the oil is carbonised, and then over a Teclu burner until the contents of the dish are white. The mass is then dissolved in hot dilute hydrochloric acid and the solution treated with barium chloride. *Free Sulphuric Acid*.—About 10 grms. of the sample are dissolved in 50 c.c. of ether, and the solution shaken with three successive portions of 50 c.c. of saturated brine of known sulphuric acid content. The united brine extracts are titrated with $\frac{N}{4}$ alkali, with methyl orange as indicator. *Alkali Salts*.—The only salts used for washing sulphonated oils are sodium sulphate and chloride, and, since the latter is converted into sulphate during the process, the salts left in the oil may be reported as Na_2SO_4 . The brine extracts from the titration are heated to boiling, the sulphate precipitated, and any free sulphuric acid present deducted. *Alkalis*.—Ten grms. of the sample are dissolved in water, and the solution titrated with $\frac{N}{2}$ acid, with methyl orange as indicator. In the absence of ammonia the result is expressed as sodium oxide. If ammonia be present, another 10 grms. of the oil are distilled with excess of sodium hydroxide, and the ammonia received in 25 c.c. of $\frac{N}{2}$ acid, and the difference between the amount of ammonia and total alkali is recorded as sodium oxide. *Unsulphonated Oil*.—A solution of about 5 grms. of the sample is neutralised with $\frac{N}{2}$ acid, and extracted with 100 c.c. of ether. The ethereal extract is repeatedly washed with water, and then shaken with 25 c.c. of water and 25 c.c. of approximately $\frac{N}{4}$ -potassium hydroxide solution, the soapy layer separated, and the ethereal layer washed with water until free from soap, the washings being united with the soap solution. The ether is then distilled and the residue of unsulphonated neutral oil dried and weighed. The water-soluble substance in the flask may be salted out and the solution shaken with a mixture of five parts (by vol.) of ether and one part of alcohol. The soap solution and washings are decomposed with acid and shaken with ether, the extract washed with water and distilled, and the residue dried and weighed. Good samples of sulphonated oil should not contain more than 15 per cent. of unsulphonated oil. A table is given showing the results thus obtained with seventeen samples of commercial sulphonated oils from castor, maize, seal and herring oils, wool grease oleine, etc.

C. A. M.

Colour Test for Oxalic Acid. L. H. Chernoff. (*J. Amer. Chem. Soc.*, 1920, **42**, 1784.)—In 5 c.c. of the solution a few crystals of resorcinol are dissolved by gentle heating and the mixture cooled. Sulphuric acid is then slowly run in to form a layer beneath the solution without the generation of much heat, when, if oxalic acid is present, a blue ring will be produced at the junction of the liquids. Should no colour be produced the contents of the tube are mixed, cooled, and 5 c.c. more of sulphuric acid added as above. If still no colour is formed the solutions are again mixed and warmed over a flame. Any blue colour produced disappears on cooling the solution in ice-water, and reappears on warming. On boiling the mixture for a few minutes, the colour becomes dark green, changing to light yellowish-green on cooling, when, if sulphuric acid is again added, the blue colour reappears. This reaction is sensitive to 1 mgrm. of oxalic acid if dissolved in a few drops of water, and if dilute solutions are to be tested it is advisable to concentrate to about 10 per cent. The reaction is unaffected by the commonly occurring organic acids, and interfering substances may be removed by precipitating the oxalic acid as the calcium salt from ammoniacal solution, washing with water, and applying the test to the aqueous suspension of the salt.

T. J. W.

Paracymene as a Solvent. A. S. Wheeler. (*J. Amer. Chem. Soc.*, 1920, **42**, 1842.)—Paracymene is a by-product of the manufacture of spruce pulp by the sulphite process, and, owing to its recent fall in price, should be available as a useful solvent, being colourless and without objectionable odour. The boiling-point is 176–176.5° C. and the refractive index 1.4905 at 12.5° C. The author describes and criticises the methods of purification, and furnishes two tables showing the solubilities at different temperatures of various organic compounds and acids. Pure paracymene remains colourless on exposure to light, but, if impure, rapidly develops a yellow or red colour.

T. J. W.

Diffusing Power of Pigments. W. K. Lewis and F. P. Baker. (*J. Ind. and Eng. Chem.*, 1920, **12**, 890-891.)—A new testing method for pigments is described, which, while not measuring the covering power, is simple both in technique and apparatus, and gives results expressed in absolute figures capable of duplication by different observers in different laboratories. If a finely divided solid be suspended in a suitable fluid, light passing through the suspension is diffused, producing a "ground glass" effect. The diffusion increases greatly with the number of particles, and is probably nearly proportional to the total number of particles, and relatively independent of their size. This diffusion is the principle employed in every turbidimeter; that is, some object is observed through the suspension, and the thickness of suspension just required to blur the object is measured. In the present method the filament of an incandescent lamp is observed, and the thickness of pigment suspension needed completely to blur the filament is determined. The suspension does not shut off the light from the filament, but the light is diffused to such a degree that the outline of the filament is no longer perceptible. The depth of suspension just necessary to obscure the filament can be determined with remarkable precision. The pigment to be tested is suspended in a suitable

fluid, and the suspension is maintained by any satisfactory method of stirring, such as using a stirring rod or blowing air through the liquid. The apparatus consists of a vertical cylinder of metal or glass, to which is cemented a plate glass bottom, plate glass being used to make sure that the two faces are absolutely parallel. The sides of the cylinder are preferably blackened, but this is not essential. The suspension, the pigment concentration of which is known, is poured into the cylinder until the filament of an incandescent lamp held just below the bottom can no longer be distinguished, and the depth of suspension is accurately noted. It has been experimentally determined that the depth of suspension observed is inversely proportional to the concentration of the pigment. This means that the disappearance of the filament is determined by the amount of pigment per unit area of cross section of the cylinder, and is uninfluenced by the position of the pigment particles in the direction in which the light is travelling. It is this weight in grams required for each square centimeter of section which is measured. The authors report the reciprocal of this quantity, *i.e.*, the sq. cs. of surface which 1 grm. of pigment is capable of covering, and this is called the diffusing power of the pigment, and, though expressed in square centimeters per gram, it will be equally satisfactory if expressed in square feet per lb. The diffusing power of a pigment is a function of the completeness with which it is dispersed in the fluid employed, and in the investigation linseed oil has usually been employed. The diffusing power must not be confused with covering power, which is unquestionably influenced by a pigment's light-absorption capacity, a factor which is entirely eliminated in the determination of diffusing power. H. F. E. H.

Determination of Sulphur Compounds in Coal. A. R. Powell. (*J. Ind. and Eng. Chem.*, 1920, 12, 887-890.)—The author has employed the methods described by Powell and Parr (University of Illinois Engineering Experiment Station, 1919, Bull. III., p. 10) with certain modifications which are now described as suitable for rapid routine work. After the sulphate and pyrites determinations are completed, the organic sulphur may be reckoned from the total sulphur by difference. The extraction of sulphate may be completed in a few minutes by boiling with hydrochloric acid (sp. gr. 1.12) rather than by slow extraction with the very dilute acid. The boiling acid does not decompose pyrites to any appreciable extent. The extraction of pyrites by dilute nitric acid may be completed in an hour or two by the use of a shaking machine. Dilute nitric acid will extract small amounts of the unstable phenol-soluble form of organic sulphur from certain coals. To avoid this error in laboratory determinations it is recommended that pyritic sulphur be calculated from the pyritic iron value. The dilute nitric acid not only extracts pyrites quantitatively but also the same iron and sulphur which is taken into solution by dilute hydrochloric acid. For this reason, samples intended for dilute nitric acid extraction need not be extracted with hydrochloric acid first. Semi-bituminous coals and the harder varieties of bituminous coals must be digested for a long time with concentrated nitric acid before the organic matter will become completely soluble in ammonia. The Eschka method is preferable to the sodium peroxide method for the determination of sulphur in coal and coal residues, unless the sodium peroxide used has a low and constant sulphur content. The following table summarises

the results of analyses of some samples of coal, and shows that all the sulphur of these coals has been accounted for in the four forms of pyrites, sulphate, and the two forms of organic sulphur.

SUMMARY OF ANALYSES FOR SULPHUR COMPOUNDS IN COAL.

Pyritic sulphur	...	...	0.47	0.79	0.08	0.13	1.75	1.99
Sulphate sulphur	...	...	0.07	0.23	0.01	0.04	0.71	0.32
Humus organic sulphur	...	...	0.50	0.46	0.44	0.42	1.01	0.39
Phenol-soluble organic sulphur	...	...	0.12	0.20	0.02	0.09	0.77	0.32
Total	...	...	1.16	1.68	0.55	0.68	4.24	3.02
Total by direct analysis	...	...	1.21	1.72	0.56	0.71	4.25	3.06
Difference between totals	...	...	0.05	0.04	0.01	0.03	0.01	0.04

H. F. E. H.

Determination of Free Sulphur and the Coefficient of Vulcanisation in Vulcanised Rubber. W. J. Kelly. (*J. Ind. and Eng. Chem.*, 1920, 12, 875-878.)—Two new methods for rubber analysis are presented; the first gives the correct value for free or elementary sulphur in rubber goods, and the second a lower and more accurate value for the coefficient of vulcanisation than is obtainable by any of the older methods. The work was all done on pure gum stock without any accelerator, and the methods described are being extended by the author to meet the conditions which obtain in compound stocks. The sulphur present in vulcanised rubber may be divided into four parts: Sulphur soluble in acetone: (1) Combined with resins and proteins, (2) true free sulphur. Sulphur insoluble in acetone: (1) Combined with rubber, (2) combined with resins and proteins. As regards the sulphur soluble in acetone it is unfortunate that all solvents for either will dissolve both to a greater or less extent. The entire extract very readily dissolves in 95 per cent. alcohol, and this alcohol will also dissolve about 0.04 gm. sulphur in 100 c.c., the amount of sulphur usually obtained in an ordinary analysis. If, however, the extract is treated with alcohol already saturated with sulphur, it will dissolve all organic material present, but none of the elementary sulphur, which remains in the flask after the solution is decanted, and can be weighed as such, or oxidised and weighed as barium sulphate. The saturated solution of sulphur in ethyl alcohol, referred to below as saturated alcohol, is therefore prepared by heating an excess of sulphur for about three hours at 70° C., and allowed to cool slowly. *Method.*—A 1-grm. sample of the ground rubber is extracted with acetone for sixteen hours in an Underwriters' apparatus. The acetone is distilled off, the residue dried at 60° to 65° C. for half an hour and treated with 50 c.c. of saturated 95 per cent. alcohol, and the flask and contents weighed. An accuracy of 0.5 gm. is sufficient for this weighing. It is then warmed to about 50° C. for a few minutes, to insure complete solution of all the organic material, and allowed to cool slowly to room temperature. Before it has reached this temperature the flask is again weighed, and any alcohol lost during the heating is replaced with pure 95 per cent. alcohol. The flask is then covered and allowed to stand for twenty-four hours, to permit

the complete crystallisation of any of the elementary sulphur of the extract which may have dissolved. The liquid is decanted and the residue washed two or three times by decantation with 5 c.c. of saturated alcohol, and dried. It may then be weighed directly, or oxidised and weighed as BaSO_4 . The figure thus obtained is the value of the true free or elemental sulphur which is available for further vulcanisation of the rubber. The remainder of the acetone-soluble sulphur is combined with resins, etc., and does not participate in further vulcanisation.

Coefficient of Vulcanisation.—A 1-grm. sample of ground rubber is extracted sixteen hours with acetone, and the residue dried. It is then boiled for eight hours with 75 c.c. of a 5 per cent. alcoholic potash solution and washed once or twice with hot alcohol. As the removal of the last traces of alkali is very difficult, the sample is extracted sixteen hours (over night) with alcohol in an Underwriters' apparatus. The solutions are then mixed, and the alcohol distilled off. The residue is oxidised first with 15 c.c. of a solution of bromine in potassium bromide (120 grms. KBr and 160 grms. Br_2 in 1 litre of water), in order to prevent the violent action of fuming nitric acid on alkali, and finished with nitric acid. The barium sulphate is precipitated in the usual manner. As there is always some silica derived from the glass, it is necessary to evaporate twice with hydrochloric acid, thoroughly dehydrate, and remove the silica before precipitating the sulphate. The figure obtained for the combined sulphur, after extraction with alcoholic potash, is much more nearly correct than any obtained by methods which do not take into account the acetone-insoluble compounds of sulphur with resins and proteins. It is still possible that there is some sulphur combined with proteins and resins, insoluble in acetone and not attacked by alcoholic potash. For the present, therefore, the figure obtained by the method presented here must be considered as a maximum value for the coefficient of vulcanisation.

H. F. E. H.

Detection of Mangrove [Tannin] in Mixed Tannin Extracts. E. Schell (*J. Soc. Leather Trades' Chem.*, 1918, **2**, 284-286; through *J. Soc. Chem. Ind.*, 1920, **39**, 496A.)—A review of the several methods suggested for the detection of mangrove in tanning extracts. The solubility of mangrove tanning in ethyl acetate is a very uncertain guide. The amount of chlorine or lime in the ash is also a doubtful indication of the presence of mangrove, since these constituents may have been introduced by using hard water. The Mulhouse dyeing test with mordanted cotton is unreliable. The precipitates obtained in the Schell test, in which a cobalt solution is added to a solution of the tanning extract, are not sufficiently well defined in colour to render it an efficient test. It can only be employed with normal extracts, and fails with "solubilised" extracts, and with those which have been subjected to a treatment with alkaline compounds and which give an alkaline reaction in solution.

INORGANIC ANALYSIS.

Estimation of Ammonia in Oil and Fat Preparations. N. Welwart. (*Chem. Zeit.*, 1920, **44**, 719.)—Certain Turkey-red oils which contain ammonia, in addition to sodium compounds, are not completely decomposed when boiled with

dilute mineral acids, and in such cases estimation of the ammonia by the usual methods gives too low results. A more accurate method is to dilute a weighed quantity of the oil preparation with water, to add a fragment of pumice and an excess of calcium chloride solution, and to distil the ammonia into standard acid, after adding excess of sodium hydroxide solution. C. A. M.

Estimation of Antimony in Lead-Antimony Alloys. L. Bertiaux. (*Ann. Chim. anal.*, 1920, 2, 273-278.)—The method depends on the titration of the antimony with potassium permanganate solution in the presence of hydrochloric acid. Poirrier's orange is added as indicator; as soon as all the antimony has been oxidised, the next drop of permanganate solution added reacts with the hydrochloric acid, liberating chlorine, which at once decolorises the indicator. The details of the method are as follows: Five grms. of the alloy (*e.g.*, antifriction metal) are dissolved by heating with a mixture of 40 c.c. of concentrated sulphuric acid and 10 grms. of potassium sulphate; after cooling, the mixture is diluted with 200 c.c. of water, 50 c.c. of hydrochloric acid, and 2 drops of a 0.1 per cent. Poirrier's orange solution are added, and the solution titrated with standardised permanganate solution. The latter must be added slowly, and the solution stirred thoroughly during the titration. Lead, tin, bismuth, copper, and arsenic do not interfere; iron, if present, is titrated together with the antimony, but it may be estimated colorimetrically in the titrated solution by means of thiocyanate and an allowance made for its quantity. W. P. S.

Estimation of Calcium and Magnesium in the Presence of Iron and Aluminium Salts. E. Canals. (*Comptes rend.*, 1920, 171, 516-518.)—On treating a solution of ferric sulphate and magnesium sulphate with sodium phosphate solution containing two drops of sulphuric acid, then rendering the liquid alkaline with ammonia, and adding acetic acid, some of the magnesium, if it exceeds a certain concentration, will be retained by the insoluble ferric phosphate. Analogous results were obtained with magnesium in the presence of aluminium salts, and with calcium in the presence of iron salts. Accurate results were obtained with quantities of 0.02 to 0.23 gm. of magnesium and 0.01 to 0.125 gm. of calcium, in the presence of iron and aluminium, by adding ammonia to the solution of the mixed salts and sodium phosphate solution until the precipitation was complete, and then adding acetic acid drop by drop, with vigorous agitation, which was continued for some minutes, until the whole of the calcium and magnesium had dissolved. C. A. M.

Indicators for Carbon Dioxide and Oxygen in Air and Flue Gas. L. H. Milligan, D. O. Crites, and W. S. Wilson. (U. S. Bureau of Mines, Tech. Paper No. 238, 1920, 24 pp.)—A portable apparatus described consists essentially of an absorption pipette connected with a graduated measuring tube. Sodium hydroxide solution (10 per cent.) is used as the absorbing liquid in the carbon dioxide apparatus and alkaline pyrogallol solution in the oxygen apparatus. The liquids in the apparatus are covered with a layer of petroleum oil to prevent absorption during the admission of the gas to be tested. Arrangements are provided for saturating the gases with moisture before they are admitted to the apparatus, for removing carbon dioxide in

the estimation of oxygen, and for cooling hot flue gases. The graduations of the scale are such that the percentage of carbon dioxide or oxygen, respectively, is indicated directly by the decrease in volume during the absorption. W. P. S.

Titration of Lead as Lead Chromate. J. M. Kolthoff. (*Pharm. Weekbl.*, 1920, 57, 934; through *Chem. Zeit. Rep.*, 1920, 44, 241.)—For small quantities of lead the neutral solution containing the metal is treated with a few drops of 5 per cent. aluminium chloride solution (to accelerate the precipitation of colloidal lead chromate), heated to 40° C., an excess of potassium chromate is added and the mixture boiled. The precipitate is collected on a filter, washed with hot water until free from potassium chromate, and then dissolved in dilute hydrochloric acid. The solution is diluted, potassium iodide is added, and the liberated iodine titrated with thiosulphate solution. Each c.c. of $\frac{N}{10}$ thiosulphate solution is equivalent to 6.9 mgrms. of lead. In the case of large amounts of lead, the solution may be treated with an excess of $\frac{N}{10}$ potassium dichromate solution; potassium iodide is then added, and the liberated iodine titrated. The error in this method amounts to about 0.15 per cent. W. P. S.

Estimation of Molybdenum. J. P. Bonardi and E. P. Barrett. (Technical Paper, No. 230, Bureau of Mines, Department of Interior, U.S.A., 1920, 35 pp.)—From a study of different methods for the estimation of molybdenum, the most satisfactory for the analysis of low-grade ores was found to be precipitation as lead molybdate, and titration with potassium permanganate after reduction of the molybdenum solution. In the volumetric method the ore is decomposed either by acid or by fusion with sodium peroxide into a soluble molybdate and insoluble compounds of the other constituents of the ore. The solution of the molybdate is filtered, acidified, and passed through a Jones "reductor" (Lord and Demorest, *Metallurgical Analysis*, 1916, 29-30), where the MoO_3 is reduced to Mo_2O_3 , which is then titrated with permanganate. In the acid method of decomposing the ore 0.5 to 5 grms. (containing not more than 0.08 gm. of molybdenum) are digested with *aqua regia*, and the liquid evaporated with sulphuric acid, and then boiled with ammonia. Copper and arsenic, if present, are removed by adding several grms. of zinc to the acid solution before passing it through the "reductor." In the decomposition by fusion with sodium peroxide a little molybdenum may be retained by the insoluble iron and copper hydroxides, and therefore in the case of high grade material the precipitate should be redissolved and again precipitated. $\frac{N}{10}$ permanganate solution is the most suitable strength for the titration, and the following amounts of ore should be taken: Five grms. of one containing less than about 3 per cent. of molybdenum (1 gm. for 15 per cent. and 0.5 gm. above 15 per cent.). If more than 16 c.c. of permanganate solution are required by a molybdenum solution no deduction is made for a blank estimation; but with a weaker molybdenum solution a small deduction is required. The volumetric method is accurate within 0.01 per cent. on the basis of a 1 gm. sample. For the gravimetric estimation the ore is decomposed with acids and treated with ammonium hydroxide. The hot solution of ammonium molybdate is treated with lead acetate in an excess of 2 to 3 c.c., the mixture heated for a few

minutes, and filtered. The precipitated lead molybdate is then ignited, cooled, and weighed. Any lead must first be removed as sulphate, and phosphorus by precipitation with magnesia mixture. Vanadium is also precipitated as lead vanadate, and a special method must be used for its separation from molybdenum. Sulphates do not interfere with the estimation. The gravimetric method is applicable to both high grade and low grade material, but is not practicable when fusion is necessary to decompose the ore.

C. A. M.

Electro-Analytical Separation of Nickel or Cobalt from Arsenic. N. H. Furman. (*J. Amer. Chem. Soc.*, 1920, 42, 1789.)—When an ammoniacal solution of a nickel salt containing an alkali arsenate is electrolysed, the nickel is deposited free from arsenic; but with a similar solution containing cobalt in place of nickel, the deposit of cobalt is invariably contaminated with arsenic. Using a mixture of the two metals no arsenic is deposited with the alloy when the ratio of cobalt to nickel is less than 1:2.5, and the total weight of the mixed metals is 0.1 gm. The method used and tested by the author was as follows: Niccolite (native nickel arsenide) was dissolved in a mixture of concentrated nitric, hydrochloric, and sulphuric acids, the excess of acid evaporated and any iron present precipitated by addition of ammonium hydroxide, the ferric hydroxide being dissolved, and the precipitation repeated until free from nickel. To about 100 c.c. of the solution 2 grms. of ammonium sulphate and 15 c.c. of ammonium hydroxide (sp. gr. 0.90) were added, and a current of 0.5 amps. with a pressure of about 3.6 volts passed from ten to twelve hours. By the use of an electrode rotating from 600 to 800 revolutions per minute and a current density of 1.25 amps. deposition is complete in about one hour. After removal of the nickel the arsenic present in the solution was estimated as magnesium pyroarsenate, or as arsenic pentasulphide. The results obtained are in excellent agreement with those given by Moore's volumetric method for nickel and cobalt.

T. J. W.

Estimation of Potassium and Sodium in the Presence of Each Other. A. Quartaroli. (*Gazz. Chim. Ital.*, 1920, 50, 64-65.)—The mixture of the chlorides of potassium and sodium is weighed, and the chlorides converted into nitrates, which are dried in a water-oven at 100° C. A small quantity of the mixture is then melted, and the solidification point of the fused mass determined. The relative proportions of potassium nitrate and sodium nitrate may be obtained directly from the result, in accordance with the following table constructed from determinations with the pure salts:

	Solidification Point. ° C.		Solidification Point. ° C.		Solidification Point. ° C.
KNO ₃ , pure ...	336	65 per cent. KNO ₃	231	30 per cent. KNO ₃	254
95 per cent. KNO ₃	312	60 " "	225	25 " "	265
90 " "	297	55 " "	220	20 " "	273
85 " "	283	50 " "	225	15 " "	280
80 " "	270	45 " "	231	10 " "	287
75 " "	255	40 " "	237	5 " "	297
70 " "	243	35 " "	245	NaNO ₃ , pure ...	306

The method is much more accurate than the usual indirect methods, since an error of 10° C. in the determination is of less importance than an error of 1 mgrm. in the estimation of the chlorine in the mixed chlorides.

C. A. M.

Volumetric Estimation of Thiocyanates by Means of Potassium Permanganate. R. Meurice. (*Ann. Chim. anal.*, 1920, 2, 272-273.)—Direct titration of thiocyanates in sulphuric acid solution with permanganate solution yields low results, but the oxidation is complete if an excess of permanganate (equal to at least one-half of the amount required for the oxidation) is added and allowed to react for a short time. The excess of permanganate may be titrated with standardised hydrogen peroxide solution.

W. P. S.

Influence of Vanadium on the Estimation of Chromium in Tungsten High-speed Steels. P. Slawik. (*Chem.-Zeit.*, 1920, 44, 633.)—During the volumetric estimation of chromium in special steels a black to red-brown coloration is produced on the addition of ferrous sulphate, which changes to yellow on back-titration with permanganate, thus confusing the end-point. This was shown to be due to the formation of complex compounds of vanadic and tungstic acids. Parallel determinations in presence and in absence respectively of tungstic acid showed that its presence does not affect the result, except that the colour at end-point is yellowish brown instead of pink.

O. E. M.

Estimation of Zirconium. M. M. Smith and C. James. (*J. Amer. Chem. Soc.*, 1920, 42, 1764.)—Zirconia ore is gradually heated with ten times its weight of potassium hydrogen fluoride until it just fuses to a clear liquid. After cooling, the melt is treated with 50 per cent. sulphuric acid, heated until dense fumes are evolved, and, when cold, dissolved in boiling water, cooled, and diluted to 250 c.c. Of this solution 100 c.c. are diluted, boiled, and precipitated with ammonium chloride and ammonium hydroxide and filtered, after which the hydroxides are washed, treated with 36 c.c. concentrated hydrochloric acid and an equal volume of water, boiled and diluted to 700 c.c. This volume is then precipitated by the addition of selenious acid solution containing approximately 12.5 per cent. of selenious acid, filtered, washed with 3 per cent. hydrochloric acid, dried, and ignited. The results obtained are in close agreement with those given by the "cupferron" method. Various modifications given in detail in the original paper are adopted in the presence of iron, titanium, and phosphoric acid, but aluminium and the rare earth metals exert no effect upon the results obtained.

T. J. W.

APPARATUS, ETC.

Chemical Analysis with Ultra-Filters. L. Moser and Kittl. (*Chem.-Zeit.*, 1920, 44, 637-638.)—The ultra-filter of Zsigmondy (*Zeitschr. anal. Chem.*, 1919, 58, 2418) is valuable in analytical work for the collection of colloidal precipitates or hydrosols, but not for general analytical work. The constant attention of the operator is necessary to avoid, on the one hand, contact of the precipitate with the

rubber joint ring, and, on the other, the running dry of the precipitate, which causes the formation of cracks through which the washing liquid passes uselessly. To remove the precipitate in the prescribed manner by means of a spatula and brush was not always possible; in the case of colloidal bismuth and silver the last traces had to be removed with moderately strong nitric acid. If the precipitate is allowed to dry it is very difficult to remove it from the filter.

O. E. M.

Apparatus for the Uniform Addition of Small Proportions of one Gas to another. R. Mezger. (*Chem.-Zeit.*, 1920, **44**, 658-659.)—Ammonia generated from slaked lime and ammonium chloride in a round flask heated by a mercury bath passes off to a mixing column filled with beads, at the bottom of which the other gas is admitted. On its way it passes a manometer and capillary. In communication with the flask is a U-tube containing mercury, which opens or closes the oblique mouth of a tube supplying gas to the burner heating the bath, so that as the pressure of the ammonia increases the flame is diminished, and *vice versa*. The capillary is chosen in accordance with a simplified form of Poiseuille's law, $V = KH$, in which V is the volume of gas passing in c.c. per second, H the difference of pressure, and K a constant for the capillary and the gas. This is determined for a given value of H , obtained by raising or lowering the oblique-ended tube, by absorbing the issuing gas in acid, titrating back, and calculating to c.c. per second. The constants of a range of tubes are determined. The method is applicable to other gases.

O. E. M.

Air-Oven Thermo-Regulator. H. M. Atkinson. (*J. Soc. Chem. Ind.*, 1920, **39**, 298t.)—A U-shaped tube has one limb connected with a bulb in the oven, whilst the other limb is closed with a stopper through which passes a short length of glass tube. The U-tube contains a quantity of mercury, and a float, having a capillary tube at the top, is placed in the limb carrying the stopper and glass tube, the capillary extending upwards into the latter. Gas is admitted through a side tube on this limb, and passes between the "shoulder" of the float and the lower end of the glass tube, and thence through the latter to the burner. As the pressure in the bulb in the oven increases, the mercury in the U-tube is gradually forced from one limb to the other, thus raising the float until the shoulder comes into contact with the end of the glass tube. The gas supply to the burner is thus shut off, but a small by-pass may be arranged by making a hole in the side of the float; the diameter of the capillary then controls the amount of gas passing through the by-pass. The lower end of the glass tube and the shoulder of the float may be ground to make a gas-tight joint, or a mercury seal can be provided at this point.

W. P. S.

Fischer's Viscosimeter. (*Chem. Zeit.*, 1920, **44**, 622.)—A new form of viscosimeter is described which is suitable for determining the viscosity of the most viscid liquids under constant conditions of temperature, etc. It consists essentially of an inner tube, at the base of which is a tap, and which is surrounded by a water-jacket similar to an upright Liebig's condenser. A thermometer is fixed in this water-jacket, and there is a second thermometer in a side tubulure which

communicates with the inner tube through the wall of the condenser. Water at the required temperature (usually 60° to 80° C.) is run through the condenser tube, and the efflux velocity of the liquid is measured by the time required for an aluminium or brass bulb to fall between two marks on the inner tube. The difference between the temperature of the heating water and of the liquid in the inner tube was found to be a constant of 4° C. For use with the aluminium bulb the apparatus is standardised upon chemically pure glycerol at 58° C., whilst, in the case of the brass bulb, the standardising solution is prepared by mixing 25 grms. of potato flour with 350 c.c. of water at 50° C., adding 50 c.c. of $\frac{N}{10}$ potassium hydroxide solution, and allowing the liquid to stand until cold. It is then heated to 70° C., introduced into the inner tube, and cooled to 58° C. by means of water in the jacket at 62° C.

C. A. M.

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REPORT.

Report on the Work of the Government Analytical Laboratory and Assay Office, Cairo, Egypt, during the Period 1913-1919. A. Lucas.—During the period covered by the Report, from 5,000 to 6,000 samples were examined annually. The analytical work comprised chiefly the examination of materials of all kinds from almost every Government Department, special war work for the Army, and investigations, etc., for the police and Ministry of Justice. There was also a considerable amount of advisory and consultative work, and during the last few years research work has taken a definite place in the programme of the Department; several papers by members of the staff of the Laboratory have been published in *THE ANALYST*. Every endeavour has been made to employ Egyptians as far as possible in the Laboratory, etc., and they now form more than 85 per cent. of the staff.

W. P. S.

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REVIEWS.

THE MANUFACTURE, REFINING AND ANALYSIS OF ANIMAL AND VEGETABLE OILS, FATS, AND WAXES, INCLUDING THE MANUFACTURE OF CANDLES, MARGARINE, AND BUTTER. By GEOFFREY MARTIN, D.Sc., Ph.D., F.I.C. London: Crosby, Lockwood and Son. Price 12s. 6d. net.

This volume, despite its somewhat ambitious title, consists of little more than 200 pages, and is stated in the Preface to be written for "practical men, industrial chemists, and manufacturers." The amount of condensation necessary to compress so extensive a subject within such limits is so extreme as seriously to prejudice its practical value. Thus, it is obviously next to hopeless to attempt, as the author does, to describe the manufacture and analysis of butter in five pages, of which two are taken up with tables of tests for colouring matters. Even more futile is it to essay the description of the chemistry and technology of margarine in seven pages, three of which are devoted to trade statistics.

It is a little difficult to see what good end can be served by this species of *précis* writing, except, indeed, as a popular exposition of the subject for the general reader, which is in this case specifically disclaimed.

One of the defects which arises from this attempt is the lack of coherence and proportion between the various sections of the subject. For example, the chapter on the hydrogenation of oils, occupying some thirty pages, is of quite disproportionate length when compared with the rest of the book. Incidentally the space in question is chiefly taken up with the text of patent specifications, without, in many cases, any accompanying explanatory matter, and without attempt at arrangement or classification. The insertion of chapters on the treatment of waste rags, and the manufacture of fish meal, is surely somewhat irrelevant, especially in view of the exclusion of so much matter of primary importance to the subject.

The portions of the book devoted to the chemistry of the oils and fats are brief and sketchy, and show the patchiness previously mentioned. Further, a good deal of the data appears to have been obtained from obsolete publications. Thus, in the two pages which are all that can apparently be spared to the chemical composition of the oils and fats, the substance *clupanodonic acid*, the characteristic fatty acid of the fish oil group, which was discovered in 1906 by Tsujimoto, is omitted from the table of fatty acids; and, although fish oils are mentioned in many places, there is apparently no reference to this distinguishing component either as fatty acid or as a glyceride.

Such an omission, although greatly to be deplored, might conceivably be due to inadvertence, but, unfortunately, there are not a few similar instances. Thus, the

table on page 4 contains "margaric acid," long ago shown to be a eutectic mixture of palmitic and stearic acids, and omits daturic acid, isolated by Meyer and Beer in 1912. Further, cerotic acid is given an odd number of carbon atoms (27 instead of 26), and several fatty acids are included which have never been obtained from natural oils and fats, while elæostearic acid is stated to have the formula $C_{19}H_{30}O_2$, whereas this was shown by Kametaka in 1903 to be erroneous.

In the section on analysis, confined to eleven pages, there are many similar instances of errors and omissions which are even less excusable. On page 151, the specific gravity of beeswax is given as lower than that of palm oil, while, further on, the "titer" of fats is defined as the *melting-point* of their fatty acids—a statement which is incidentally contradicted on the same page.

After all that has been written on the desirability of a uniform temperature for recording the refractive index of oils and fats, resulting in the virtually unanimous agreement of the figure of 40° C. as the most suitable, the statement is made on page 152 that "the refractive index of oils is usually determined at 60° C."

Similarly in the description of the Polenske value—erroneously spelt with an acute accent over the final "e"—one is still directed to "take a few fragments of pumice," notwithstanding that this very statement has been pilloried many times in this journal and elsewhere.

On page 162 are given percentage yields of insoluble bromides from oils without any mention of the conditions under which these were obtained, which, of course, vitally affect the figures. Similarly, the Maumené test is stated to be made with "concentrated sulphuric acid," with no further indication of strength.

In spite of much recent work on the "Valenta test," this is dismissed in two or three lines as a test for butter only, no mention being made of its general application. Further, the strength of the acetic acid on which the temperature figure entirely depends is not mentioned otherwise than as "glacial," which has been shown to be useless as a definition for this test.

In the section on butter, no mention is made of the Kirschner or even of the Polenske test in determining the purity of butter fat, but it is stated that the iodine value is "particularly useful in detecting adulteration with coconut fat."

There are several misprints and other signs of hurried proof correction, but these are of small moment compared with the foregoing inaccuracies.

Although, therefore, the book may present features of interest to the technical reader, it is scarcely possible to recommend it from the chemical and analytical standpoint.

P. J. FRYER.

DICTIONARY OF EXPLOSIVES. By ARTHUR MARSHALL. Pp. xiv+159. London: J. and A. Churchill, 1920. Price 15s. net.

The present volume is Mr. Marshall's third publication on the subject of explosives. It is a careful compilation on the composition, and certain of the properties, of about four hundred explosives. The subject-matter is restricted almost entirely to industrial explosives, which are more or less complex mixtures. The military explosives, which are either single chemical compounds or simple mixtures, are dealt with at length in Mr. Marshall's other works.

Explosives of foreign origin are included, but the explosives described are almost entirely those which are or have been in practical use. The many hundreds which have been proposed, but have failed to pass the test of actual performance, are not mentioned unless of special historical interest.

The dictionary is divided into three sections. The first contains a classification of explosives according to the purposes for which they are intended to be used; the second, which constitutes the principal part of the book, gives short descriptions of the explosives arranged in alphabetical order; and the third section is a list of the separate ingredients, with an indication of the explosives in which each occurs.

Many of the descriptions are necessarily somewhat vague, on account of the reticence of manufacturers to disclose the composition of their products. This is especially noticeable in regard to the American explosives, for many of which the only information available is a statement of the type to which they belong.

Within its somewhat restricted range, the volume contains a large amount of information, and should prove of considerable value as a work of reference. It is well printed, and no typographical errors have been observed. The style of binding is, however, not such as to produce a good impression, and the price seems very high for the size of the book.

W. L. TURNER.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

ORDINARY MEETING. NOVEMBER 3, 1920.

HELD at the Chemical Society's Rooms, Burlington House, Mr. Alfred Smetham, President, in the chair.

Certificates were read for the first time in favour of Messrs. Urban Aspey, Herbert Corner Reynard, B.Sc. (London), A.I.C., Edwin Burnhope Hughes, B.Sc. (London), A.I.C., Harry Jephcott, M.Sc. (London), A.I.C., Arnold Lees, A.I.C.

A Certificate was read for the second time in favour of Mr. T. K. Ghose, B.A.

The following was elected a Member of the Society: Mr. Udolphus Aylmer Coates.

The following papers were read: "The Gravimetric Estimation of Bismuth as Phosphate and its Applications in Ore Analysis," by W. R. Schoeller, Ph.D., and E. F. Waterhouse; "The Time Factor in Saponification," by Percival J. Fryer, F.I.C.; "The Position of Analytical Chemistry in France," by Victor Cofman, B.Sc.; "Apparatus for Collecting Samples of Water at Great Depths," by W. T. Burgess, F.I.C.



THE GRAVIMETRIC ESTIMATION OF BISMUTH AS PHOSPHATE AND ITS APPLICATION IN ORE ANALYSIS.

By W. R. SCHOELLER, PH.D., AND E. F. WATERHOUSE.

(Read at the Meeting, November 3, 1920.)

THIS paper is the result of an investigation into the analytical chemistry of bismuth, in the course of which we elaborated a slightly modified method for estimating the metal as phosphate. As pointed out by Moser in his monograph, "*Die Bestimmungsmethoden des Wismuths*" (Stuttgart, 1909), the phosphate process is the most advantageous form of bismuth estimation; bismuth phosphate, BiPO_4 , is of definite composition; it forms a white, heavy, crystalline precipitate resembling lead sulphate, quite insoluble in water and in very dilute nitric acid, depositing and filtering quickly,

unchanged by ignition, and not readily reduced. In spite of these advantages, the method does not appear to have found a place in any English textbook. It has been studied by Stähler and Scharfenberg (*Ber.*, 1905, 38, 3862), Salkowski (*ibid.*, 38, 3943), Moser (*Zeitsch. anal. Chem.*, 1906, 45, 19), and Stähler (*Chem. Zeit.*, 1907, 31, 615). Salkowski adds 5 c.c. of 25 per cent. phosphoric acid to the hot, moderately acid nitrate solution free from hydrochloric acid or chlorides, which have a solvent effect on bismuth phosphate. Stähler and Scharfenberg use a 10 per cent. solution of trisodium phosphate, enough of which is added to neutralise nearly all the nitric acid present in the solution; they state that disodium phosphate may also be used, but in this case the final volume of the liquid, if rather acid at the start, may become inconveniently large. These directions make it plain that a very large excess of precipitant is used, especially as chlorides are stated not to interfere provided sufficient sodium phosphate is added. Moser, on the other hand, adds only a very slight excess of $\frac{N}{2}$ diammonium phosphate solution to the liquid containing no chlorides and as little free nitric acid as possible. Stähler almost neutralises the nitrate solution with trisodium phosphate, using rosolic acid as an indicator. This very brief survey will suffice to show a marked variation in the procedure adopted by different investigators for the precipitation of bismuth phosphate; moreover, their views as to the solubility of the precipitate in dilute nitric acid would appear not to be the same, and some of the directions for adjusting the acidity of the solution are sufficiently vague to make experimentation necessary before confidence in the accuracy of the process is finally gained. We felt, therefore, that it might be useful to repeat the earlier work and, if possible, improve upon it. Working with pure bismuth nitrate solutions, we soon arrived at the conclusion that the quantitative precipitation of the phosphate was a matter of rather delicate adjustment: if the acidity was kept low, the liquid on being heated to boiling usually deposited a heavy crystalline precipitate of oxynitrate, to re-dissolve which it was necessary either to add a disproportionate amount of nitric acid or evaporate to small bulk. Again, the phosphate obtained from solutions containing but little nitric acid was rather bulky and flocculent, and appeared to lead to slightly high results. If, on the other hand, the acidity was a little higher than necessary to prevent deposition of the subnitrate, a small fraction of the bismuth failed to precipitate as phosphate unless an excessive quantity of alkaline phosphate was added. This fact would explain the use of trisodium phosphate by Stähler and Scharfenberg. As regards Moser's practice—i.e., addition of a slight excess of ammonium phosphate over that required to react with the bismuth nitrate—we were unable, as a rule, thereby to effect complete precipitation. Further work led to the adoption of our modified process, which is free from the drawbacks indicated above, and has given consistently good results; the directions about to be given are precise enough to enable those unfamiliar with the method to apply it successfully.

Authors' Method for Estimating Bismuth as Phosphate.—The cold bismuth solution, containing no chlorides and an indefinite amount of nitric acid in a total bulk of less than 100 c.c., is carefully treated with strong ammonia until a slight permanent precipitate is obtained. The precipitate, being formed in the cold, is not the crystalline oxynitrate, but a flocculent basic nitrate which dissolves at once on addi-

tion of 2 c.c. of strong nitric acid. The clear liquid is now heated to boiling and precipitated whilst boiling with a 10 per cent. diammonium phosphate solution contained in a burette. The addition should proceed very slowly at first (about 30 drops per minute) to favour the formation of a coarsely-crystalline precipitate; once the bismuth phosphate is thrown down, the remainder of the precipitant may be added more rapidly. It is advisable to stir the solution, as the beaker is liable to bump badly. A considerable excess of ammonium phosphate should be used, viz.:

20	c.c. of 10 per cent. solution for	0.05	gram. bismuth.
30	"	"	0.1
40	"	"	0.2
50	"	"	0.3
60	"	"	0.4 to 0.5

When the required amount of precipitant has been added, the solution is diluted to 300—400 c.c. with boiling water and left to settle for ten to fifteen minutes on a hot plate or water-bath. The clear liquid is then decanted through a 9 cm. No. 40 Whatman filter, or preferably a Gooch crucible; the precipitate is stirred up twice with a hot 3 per cent. ammonium nitrate solution containing a few drops of nitric acid per litre, transferred to the filter, the washing completed, and the precipitate dried and ignited gently. If a filter paper is used, the precipitate is detached as completely as possible, and the paper charred at low temperature in a porcelain crucible. Factor $\text{Bi} : \text{BiPO}_4 = 0.6865$.

The method in its present form entirely obviates the formation of crystalline oxynitrate caused by deficiency of nitric acid, while the subsequent dilution of the liquid reduces the concentration of the nitric acid to a point where the last traces of the bismuth are precipitated by the excess of ammonium phosphate. The filtrate from the bismuth phosphate usually remains quite colourless on treatment with hydrogen sulphide; sometimes a faint coloration is produced, but the loss is altogether negligible (*i.e.*, less than 0.0002 gram.).

Authors' Process for Estimating Bismuth in Ores.—This process, which we are using in actual practice, consists in determining bismuth as phosphate after separating it from lead, arsenic, antimony, copper, and iron. Special attention is given to the quantitative separation of lead, which we effect by Clark's method, *i.e.*, precipitation of the bismuth by metallic iron, lead (and any tin present) remaining in solution (*J. Soc. Chem. Ind.*, 1900, 19, 26). This is undoubtedly the most satisfactory practical method for the separation of the two metals. Galletly and Henderson, who tested it, found it to be "not only easily carried out, but also capable of yielding accurate results" (*ANALYST*, 1909, 34, 389); but, like the phosphate method for bismuth, it has not yet found its way into any of the standard textbooks. The separation of arsenic and antimony is carried out, as usual, by digesting the hydrogen sulphide precipitate with alkaline hydroxide and sulphide, any molybdenum present being also extracted. If the ore carries copper, the precipitated sulphides are digested with sodium cyanide, which dissolves copper sulphide; arsenic, antimony, and copper may be removed in one operation. Mercury, a rare constituent, is eliminated when the sulphides are treated with

nitric acid, in which the mercury compound is insoluble. The assay is carried out as follows :

The ore (1 grm. or more, according to the bismuth content) is digested on a hot plate with strong hydrochloric acid ; galena, if present, is thus decomposed without formation of lead sulphate. Nitric acid is then added, and the assay evaporated almost to dryness. The nitrates are again converted into chlorides by evaporation with hydrochloric acid ; the residue is taken up with the same acid, and after moderate dilution with water, the gangue is filtered off and washed with dilute hydrochloric acid (1 : 10 water). The filtrate is precipitated with 1 to 2 grms. of pure, very fine iron wire (one foot of which weighs about 0.1 grm.), the action being allowed to proceed at boiling heat for ten to twenty minutes. The metallic sponge and excess iron are filtered off, washed with boiling water, returned to the beaker, and dissolved in hot hydrochloric acid and a few drops of bromine. When the metals have dissolved the solution is boiled till bromine is expelled, diluted, precipitated with hydrogen sulphide, and the precipitate filtered off and washed with acidulated hydrogen sulphide water. The sulphides are returned to the beaker and treated hot with freshly-made caustic soda and hydrogen sulphide, a freshly-made solution of sodium cyanide being added if copper is present. The residue from this extraction is filtered on the paper previously used, and washed with dilute sodium sulphide solution. The filter is then spread against the side of the beaker, the precipitate rinsed down, and the paper cleaned with hot dilute nitric acid (1 : 1). The washed filter is discarded, more nitric acid added, and the assay evaporated until the sulphur has fused into a transparent globule. Bromine should on no account be used at this stage, or low results may ensue ; if nitric acid alone fails to decompose the sulphide completely, the presence of mercury is indicated. The sulphur is filtered off on a very small filter which is washed with dilute nitric acid ; the filtrate, which should occupy less than 100 c.c., is now ready for the phosphate precipitation.

If a preliminary test proves lead to be absent, the iron precipitation is omitted and the filtrate from the gangue treated with hydrogen sulphide. If copper and the metals of the arsenic sub-group are known to be absent, the hydrogen sulphide precipitate is dissolved at once in nitric acid for the precipitation of the bismuth as phosphate.

Results of Test Analyses.—A number of ores were assayed by us according to the method described above, with the following results :

Ore A contained As, Sb, and a little Pb.

Bi (as BiOCl) : 4.63 ; 4.62 per cent.

Bi (as BiPO₄) : 4.64 ; 4.70 ; 4.72 per cent.

Ore B contained Pb, Cu, and As.

Bi (as BiOCl) : 15.83 ; 15.70 ; 15.71 per cent.

Bi (as BiPO₄) : 15.76 ; 15.82 ; 15.83 ; 15.86 per cent.

Ore C contained wolfram and very little copper. The filtrate from the gangue was treated with hydrogen sulphide, the precipitate dissolved in nitric acid, and the solution precipitated with ammonium phosphate.

Bi (as BiOCl): 34.15 per cent.

Bi (as BiPO_4): 34.05; 34.09; 34.03; 34.17 per cent.

Ore D contained small amounts of As, Sb, and Pb.

Bi (as BiOCl): 20.17 per cent.

Bi (as BiPO_4): 20.01; 20.05; 20.21; 20.13 per cent.

Summary and Conclusion.—A modification of the method for the gravimetric estimation of bismuth as phosphate is described, the directions being made sufficiently explicit to render the method easily applicable even if the operator has only an occasional determination to make. A process for the estimation of bismuth in ores is also described in detail; in this the bismuth is removed by iron wire, copper, arsenic, and antimony by extraction of the sulphides with sodium cyanide and sulphide, and the bismuth sulphide converted into and weighed as phosphate.

As a result of our experience with the above process we endorse Moser's opinion of the phosphate method—viz., that in the great majority of cases it should prove the most advantageous method, and be accorded first place in the textbooks.

THE SIR JOHN CASS TECHNICAL INSTITUTE,
ALDGATE.

DISCUSSION.

Mr. C. E. BARRS said he had considerable experience in the analysis of bismuth in metals and ores, usually carried out by weighing on a tared filter paper as bismuth oxychloride.

In the first place, it should be remembered that bismuth ores are often very complex, containing most of the common metals, and sometimes silver, gold, tungsten, molybdenum, tellurium, etc. He had had an opportunity of trying the separation of the bismuth with iron wire, and found that if carried out as directed, it is very useful as a satisfactory separation from lead and tin, both of which may lead to inaccuracies, whether the bismuth is estimated as phosphate or oxychloride.

The estimation of small quantities of bismuth—say, under 0.25 per cent.—may be made colorimetrically by precipitation as oxychloride, with subsequent solution of the precipitate in sulphuric acid, and dilution to known volume, *small* portions being taken, with the usual addition of potassium iodide, for the colour test in order that the tint shall not be too strong.

He asked the authors what their experience had been when treating the sulphuretted hydrogen precipitate with cyanide. He had found that in some cases, and more particularly if tin is present, a tendency for the bismuth sulphide to wash through the filter, but this seems to be prevented if the precipitate is given a final wash with hot water before washing back into the beaker for digestion with cyanide.



THE POSITION OF ANALYTICAL CHEMISTRY IN FRANCE.

BY L. COFMAN, B.Sc.

(Read at the Meeting, November 3, 1920.)

LET me say at once that the present article deals solely with the professional side of the subject. Information regarding recent development and progress in Analytical Chemistry can be gathered easily from periodicals devoted to it, but one looks in vain for enlightenment concerning the conditions under which the profession is carried on. Such conditions are, nevertheless, important, even from a purely scientific point of view, for they react on the quantity and quality of original work done, especially in a branch of science where the bulk of the research is done by the rank and file.

EDUCATION.

One of the chief drawbacks to a satisfactory organisation of the analytical profession in France is the lack of an institution devoted entirely, or mainly, to its teaching. Chemistry is taught in the Science Departments and in a number of Institutes of Applied Chemistry attached to various Universities throughout the country. Such are the Institutes of Applied Chemistry of Paris, Lyon, Nancy, Lille, Toulouse, Clermont-Ferrand, the Electrochemical Institute of Grenoble, and the School of Chemistry of Bordeaux. Training in Analytical Chemistry is also given in the Higher Schools of Pharmacy and in the Municipal School of Physics and Chemistry of Paris. The Pasteur Institute, the National Agronomic Institute, and various other bodies have courses devoted to more or less specialised branches of Chemistry.

The training received by the student naturally varies greatly from place to place. At the Institute de Chimie Appliquée of Paris, a three years' course is provided, at the end of which a degree of "ingénieur-chimiste" is conferred on the candidate who has successfully passed the examinations, one at the end of each year. This Institute will no doubt play in future a more important part than it has done so far in chemical education. The premises which it occupies at present bear a not very flattering testimony to the esteem in which Applied Chemistry was held in France before the war. Students and apparatus are crowded together in a set of irregular buildings entirely unsuited for such use. Fortunately the lessons of the great war have not been all unheeded. The University of Paris, realising the great importance of Applied Chemistry in modern life, has decided to move the Institute from its old site at 3, Rue Michelet, to more spacious premises at the centre of the University grounds, Rue Pierre Curie. The new building is rapidly being completed, and provided with all necessary modern appliances and with every comfort for the students.

THE TITLES OF "CHIMISTE-ANALYSTE," "CHIMISTE-EXPERT," AND
"PHARMACIEN-CHIMISTE."

None of the educational bodies so far mentioned confers a degree in analytical chemistry, and the title of "chimiste-analyste" is fully as much abused in France as its equivalent is in England. The profession includes :

- (i.) Chemists employed in the various laboratories in connection with the repression of frauds, or who occupy other official positions.
- (ii.) Those placed on the list of experts to the Courts of Justice, who may be called as advisers in chemical matters.
- (iii.) Analytical and consulting chemists with private laboratories, of whom there are relatively few.
- (iv.) Pharmacists in business who undertake certain kinds of analytical work, especially of a biological character.

Following the insistent efforts of M. Jazeneuve, a law was passed in 1913* creating a title of "chimiste-expert du Government." The jury examining for this qualification is composed of teachers from the higher colleges which impart chemical training, and of members of a Permanent Technical Committee attached to the Ministry of Industry and Commerce. The law provides that special courses in analytical chemistry shall be instituted at certain Universities for training candidates already possessing an advanced knowledge of chemistry. The "chimistes-experts" exercising the profession previous to the legislation could obtain the new title on showing that they possessed an adequate qualification.

A recent bye-law (June, 1920) creates a post of "pharmacien-chimiste" in the Army, obtained by competition before an examining jury. The duties of the pharmacien-chimiste in peace-time include the analysis of foods and water supplies, and the assay of drugs and chemicals intended for military hospitals. In time of war they would be placed in charge of the Army Chemical and Toxicological Laboratories.

PROFESSIONAL ASSOCIATIONS, PERIODICALS.

There is much similarity between the French and the English chemical associations. Thus the Société des Chimistes-Experts de France corresponds to the Society of Public Analysts and other Analytical Chemists, and the Société des Chimistes Français has more than one point in common with the Institute of Chemistry as modified of late.

The first-mentioned Society aims at bringing together all those who are, or intend to be, nominated as "chimistes-experts" to the Courts of Law. Its official organ is the *Annales des Falsifications*. No special qualifications are required for membership. The Society was formed in 1912, and two years later had acquired over 500 members (the total number of chemists inscribed on the expert-lists is about 1,000). The following list gives the occupation of the members, and is interesting as showing approximately the proportion of different kinds of "analytical chemists."

* This Act, pending the promulgation of further bye-laws and regulations, has remained without practical application so far.

Government officials (chemists employed in municipal laboratories, etc.)	187
Military (pharmacists in the Army, Navy, and Colonies)...	66
Chemists, free	79
Pharmacists, civil	153
Various (industrials, doctors, engineers)	26

Total 511

The other and more recent Association, the Société des Chimistes Français, was intended to include all chemists of French nationality. It is divided into an industrial and an analytical section, the members being further classed in different categories according to qualification and experience possessed. The *Annales de Chimie Analytique* has become the organ of the Society, which has at present some 460 members. Both the above societies are making numerous efforts to improve the social and material conditions of the analytical chemists.

THE ORGANISATION IN CONNECTION WITH THE REPRESSION OF FRAUDS.

Special legislation for the inspection of pharmaceutical and allied products existed in France at an early period (law of 21 Germinal, year XI.), but the first important modern legislation for the protection of the public from adulterated food was passed in 1905. This and subsequent bye-laws instituted various services for the purpose. The inspection of foodstuffs, beverages, and products for agricultural use is entrusted to officials nominated by the prefect (the county representative of the Home Office). These officials, of which there are some 1,000 throughout the country, occupy the ranks of police inspector, veterinary, county, or municipal agents. Their payment consists of a fixed small fee per sample taken in addition to travelling expenses. A certain number of Syndicates provide the authorities with experienced agents at their own expense. A chief inspector and fourteen county inspectors are charged with the control of this organisation.

Of the four identical samples sealed by the agent at an inspection, three are retained for subsequent reference and one is forwarded to an official laboratory. There are forty such laboratories maintained by different towns or counties. The Government also contributes to their upkeep by an amount depending on the number of samples analysed annually. This is fixed at 1.75 analyses per 1,000 inhabitants, which gives a total of 70,000 analyses carried out yearly in the whole of France.

The most important of the above-mentioned laboratories is the Laboratoire Centrale d'Analyse des Matières Alimentaires, of Paris. It is almost elegantly maintained, and consists of seven principal laboratories: two of these are devoted to the analysis of wine, carried out in a very thorough manner; two others are allotted to oils and fats, and the rest to other articles of food. An important part of the work consists in the testing of new analytical methods. If these are found more reliable than the ones already in use, circulars are sent out rendering their employment compulsory in all official analyses.

For the analysis of certain classes of products there exist special laboratories; for instance, the Laboratoire des Produits Résineux at the University of Bordeaux and

the Laboratoire des Conserve de l'Armée, the latter for the supervision of preserved meat and fish.

The surveyance of drugs and similar products is quite distinct from that of food. Pharmacies, dispensaries, and mineral water factories must be inspected yearly by a pharmacist nominated by the prefect on the recommendation of the local Faculty, or School, of Pharmacy. Drug stores, perfumeries, and other premises where medicinal or hygienic preparations are sold, are also subject to inspection. The analysis of suspected materials is carried out in laboratories attached to the Schools of Pharmacy, but placed under the supervision of the Ministry of Agriculture. Twenty such laboratories exist in France. The one in Paris is naturally the better organised. In addition to the analysis of drugs and chemicals there is a bacteriological section for the testing of the germicidal power of disinfectants, the adequate sterilisation of surgical dressings, etc. Further, a large amount of research is always in progress.

OTHER LABORATORIES.

The Municipal Laboratory, attached to the Prefecture de Police of Paris, devotes much of its activity to food analysis, biological examinations, and physiological testing for private individuals, at a nominal fee. It possesses more extensive accommodation, but is less conveniently arranged than either of the previously described laboratories.

At the Institute des Arts et Métiers there is a Department corresponding to the National Physical Laboratory of Teddington ; its Chemical Section undertakes the testing and assaying of lubricating oils, alloys, and other industrial products.

Finally may be mentioned the Laboratoire d'Essais de Semences for the testing of seeds, and the Institute Pasteur for biological examinations.

It will be seen from the above-outlined description of French institutions that the work carried out in England by the Public Analyst is divided in France amongst several organisations. This division of labour has its disadvantages, and overlapping cannot be avoided, for instance, in the case of substances, such as olive oil, which are used both as food and for pharmaceutical purposes. But one undoubtedly good point is the official testing and unification of analytical methods, which would render impossible the promulgation of orders or regulations which cannot be carried out such as the recent Cocoa Order of the Food Controller, which set a limit to the amount of shell allowable, without indicating the method to be followed for its estimation.

DISCUSSION.

Mr. CRIBB said that the Report of the Eighth International Congress of Applied Chemistry contained a series of papers on the methods adopted for dealing with adulteration in Canada, the United States, and most of the Western European nations. From Mr. Cofman's paper it would appear that no noteworthy advances had been made in France, either in legislation or administration, since the date of that Congress.

The most important of the French laws relating to adulteration were, however, passed as recently as 1905, and, as there was a central executive, more likely to be

better and more uniformly administered than was possible with the worn-out laws in force in this country.

In two directions the French system was ahead of ours. They could take samples from the wholesale dealer and manufacturer, and the judge was allowed the assistance of a scientific expert in difficult cases. There was some reason for hoping that before long our more or less obsolete legislation would receive the attention of the Government, and such papers as that of Mr. Cofman and those to which he referred, would afford very useful suggestions.



STATUTORY STANDARD FOR GHEE.

By T. K. GHOSE.

IN view of the fact that ghee is an important article in the Indian dietary, and that it is difficult to purchase good ghee at the present time, it has become necessary to fix a standard for the genuine article. Ghee may be described as clarified butter—*i.e.*, butter fat from which moisture, casein, and other constituents, except butter fat, have been removed. The literature regarding the composition of ghee is very meagre. European textbooks deal with butter fat, whereas Indian ghee consists essentially of fat from buffalo milk, though there may be a slight admixture of butter from cow's milk. Richmond, Bolton, Revis, Trimen, and others have published work on ghee (*Analyst*, 1910, **35**, 343; 1911, **36**, 392; 1913, **38**, 242).

Investigations into the composition of Indian ghee have been carried on in the Corporation Laboratory for a number of years. Genuine samples of ghee, derived from individual animals as well as from herds, have been analysed, and the results recorded in Tables I. and II. Altogether 67 samples from individual buffaloes and 66 samples from herds were analysed. The minimum Reichert-Wollny value obtained was 29.2 and the maximum 42.0, the average being 34.5. The minimum of 29.2 was obtained in only one case out of 233, the next minimum being 30. In judging the quality of a commercial sample of ghee the Reichert-Wollny value 30 was adopted as the standard for purity. In October, 1918, a conference of the chemists of Calcutta was held, and the question of a standard for ghee discussed. It was decided that a sample of ghee would be declared as pure when the Reichert-Wollny value was 30 and above, and no sample would be regarded as adulterated under the Act unless it was below 28, and provided the other tests corroborated. The Corporation standard of 30 was reduced to 28, at the suggestion of one of the members of the conference, in consideration of accidental admixture with cows' ghee. It may be stated here that the English standard for butter fat, adopted by the Ministry of Agriculture and the Society of Public Analysts, is 24, though no statutory standard has been laid down.

TABLE I.

Results of Analysis of Samples of Buffalo Ghee made in the Laboratory from Milk of single Buffaloes.

Serial Number.	M.-pt.	Reichert-Wollny Value.	Saponification Value.	Serial Number.	M.-pt.	Reichert-Wollny Value.	Saponification Value.
1	35.5	38.6	237	35	36	35.8	236
2	35.5	39.3	238	36	37	30	230
3	36.5	37.5	235	37	37	33	235
4	36.8	31.8	230	38	36	35.8	237
5	34	31.5	230	39	36	38.8	237
6	35.3	34.5	234	40	36	38.9	239
7	36	34.6	233	41	37	37	236
8	37.3	35.4	234	42	36	35.6	235
9	36	30.5	229	43	36.5	34.3	232
10	35.5	33.1	231	44	37	32.7	231
11	38	33.4	232	45	36	36.2	235
12	36	42	239	46	36	35.9	236
13	37	36.2	236	47	35	40.6	240
14	35	39.9	239	48	35	39.9	240
15	35	41.9	238	49	35	39.2	238
16	34	40.6	238	50	35.5	39.5	239
17	34	39.1	237	51	36	37	238
18	35	30	229	52	36	36.9	234
19	36	34.8	234	53	36	34.3	233
20	35	42	240	54	37	34.6	233
21	35	36.2	236	55	36	34.5	234
22	36	31.3	230	56	35	36.5	235
23	36	33	231	57	35	35.2	235
24	37	34.6	232	58	35.5	36.1	236
25	37	34	233	59	36	34.1	235
26	36	35.7	235	60	34.5	38.7	236
27	36	35.7	234	61	36.5	31.7	230
28	37	30.4	227	62	36	34.5	233
29	35	32	233	63	36	34.8	232
30	36	31.8	231	64	37	30.1	227
31	36	31	231	65	37	30.2	226
32	36	32.7	232	66	36	30.8	229
33	37	36.1	235	67	36	30.8	227
34	36	37.2	237				

TABLE II.

Results of Analysis of Samples of Buffalo Ghee made from Butter produced from Mixed Milk of several Buffaloes. The Samples were collected from Dairies in different parts of India.

Serial Number.	Butyro Refractometer Reading at 40° C.	Reichert-Wollny Value.	Saponification Value.	Serial Number.	Butyro Refractometer Reading at 40° C.	Reichert-Wollny Value.	Saponification Value.
1	—	36.1	234	43	42	34.7	233
2	—	38	236	44	42	36.3	233
3	—	35	233	45	42	32.5	230
4	—	39	238	46	42	33.9	231
5	—	36	236	47	41	34.9	231
6	42	30.1	228	48	41	34.1	230
7	42	32.3	233	49	41.5	34.4	232
8	42	36.1	236	50	41	34.5	234
9	42	33.2	230	51	42	33.2	232
10	42	34.9	232	52	41	35.1	234
11	42	33	233	53	41	35	230
12	42	31.9	230	54	41.5	35	235
13	42	36.3	235	55	42	32.6	230
14	42	32.8	230	56	42	33.7	231
15	41.5	36.2	234	57	41	36.1	232
16	41.5	35.1	236	58	41	35.9	230
17	41	34.6	234	59	41	36	233
18	41.5	35	233	60	42	31.1	230
19	42	31.1	229	61	42	31.2	229
20	42	32.6	230	62	42	30.4	229
21	41.5	35.7	232	63	42	30.6	228
22	42	33.6	231	64	42	32.6	230
23	42	33.7	230	65	42	31.1	229
24	42	32.7	230	66	42	30	230
25	42	29.8	228	67	42	33.8	231
26	42	31.5	229	68	42	33.2	231
27	42	34.8	231	69	41	36.7	234
28	41.5	34.9	232	70	41	33.9	232
29	42	34	232	71	41	35.6	234
30	42	34	230	72	41	33.9	231
31	42	35.1	230	73	42	32.5	231
32	42	36	233	74	42	33.6	232
33	42	34.9	231	75	42	30.5	229
34	42	32.8	229	76	42	35.4	231
35	41.5	36.1	234	77	42	32.1	230
36	42	32.1	230	78	42	30	228
37	41.5	34.4	230	79	42	33.4	230
38	41.5	35.6	231	80	42	35.2	230
39	42	34.4	230	81	42	31.7	231
40	42	34.4	232	82	42	35.7	233
41	42	33.5	230	83	42	37.8	236
42	42	34	236	84	41	35.6	233

Serial Number.	Butyro Re-fractometer Reading at 40° C.	Reichert-Wollny Value.	Saponification Value.	Serial Number.	Butyro Re-fractometer Reading at 40° C.	Reichert-Wollny Value.	Saponification Value.
85	41	35.3	235	126	42	32.9	230
86	41	36	233	127	41.5	30.5	229
87	41	33	230	128	41	30.9	228
88	42	34.1	231	129	42	30.5	227
89	42	36.2	232	130	41	32.1	230
90	42	32.6	230	131	42	31.3	230
91	42	35	234	132	42	29	227
92	42	30.1	229	133	42	30	229
93	40	30.7	230	134	41	31.2	230
94	42	36.3	235	135	42	31	230
95	41	35	232	136	42	31	229
96	41	37.6	236	137	42	30.3	227
97	41	36.7	236	138	40	31	230
98	42	32.8	230	139	42	31	220
99	42	32	230	140	42	31	230
100	42	38.2	236	141	42	30.9	227
101	40	29.9	228	142	41.5	31.5	229
102	42	29.6	227	143	42	31.5	228
103	42	29.4	228	144	42	30.5	229
104	42	29	227	145	42	32	230
105	40	32.6	230	146	40	31	229
106	42	33.3	232	147	41	32.3	230
107	41	29.2	230	148	41	34.6	231
108	42	29.1	227	149	41	36	231
109	40	31.3	229	150	42	30	227
110	42	29.6	228	151	42	31	230
111	42	32.1	231	152	42	34.8	233
112	42	32.2	231	153	40	31.9	230
113	42	31	230	154	42	32.3	231
114	42	32.8	230	155	42	32.8	230
115	42	34.5	232	156	42	33	233
116	42	35.4	234	157	41.5	30.4	227
117	42	33.9	233	158	42	29.2	226
118	42	33.3	232	159	42	30	226
119	42	32	230	160	42	30.6	228
120	40	32.3	230	161	42	30.5	227
121	42	30.1	229	162	42	31.8	227
122	42	32.6	230	163	42	30	226
123	42	32	229	164	41.5	31.8	228
124	41.5	31	230	165	41.5	30.5	226
125	42	32.6	231	166	42	30.7	226

NOTE.

The Editor desires to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

LOSS OF SPIRIT STRENGTH DUE TO EVAPORATION.

The following figures were obtained by measuring the loss of alcohol in spirit kept in a bottle lightly covered with a watch-glass, and having an exposed surface area of approximately 1 square inch. The determinations were made with spirit kept at 65° F. and 80° F. respectively, and the sp. gr. determined in each case at 60° F., the total volume of spirit being about 200 c.c.

65° F.					80° F.				
Date.	Sp. Gr.	Proof Spirit.	Loss.		Sp. Gr.	Proof Spirit.	Loss.		
		Per Cent.	Per Cent.			Per Cent.	Per Cent.		
16/4/20	954.05	= 67.42	—		954.05	= 67.42	—		
17/4/20	954.35	= 67.07	0.35		954.6	= 66.77	0.65		
19/4/20	954.4	= 67.01	0.06		954.7	= 66.65	0.12		
20/4/20	954.5	= 66.89	0.12		955.0	= 66.30	0.35		
21/4/20	954.6	= 66.77	0.12		955.3	= 65.94	0.36		
22/4/20	954.9	= 66.42	0.35		955.6	= 65.58	0.36		
23/4/20	955.1	= 66.18	0.24		955.9	= 65.21	0.37		
26/4/20	955.5	= 65.70	0.48		956.2	= 64.85	0.36		
27/4/20	955.7	= 65.46	0.24		956.5	= 64.48	0.37		
28/4/20	955.75	= 65.40	0.06		957.0	= 63.86	0.62		
29/4/20	955.8	= 65.33	0.07		957.5	= 63.23	0.63		
30/4/20	955.9	= 65.21	0.12		957.7	= 62.98	0.25		
1/5/20	956.2	= 64.85	0.36		958.2	= 62.35	0.63		
3/5/20	956.6	= 64.36	0.49		958.7	= 61.71	0.64		
4/5/20	956.8	= 64.11	0.25		959.2	= 61.07	0.64		
5/5/20	957.15	= 63.67	0.44		959.5	= 60.68	0.39		
6/5/20	957.5	= 63.23	0.44		959.8	= 60.29	0.39		
Total loss in twenty days			4.19		Total loss in twenty days			7.13	

L. BRIANT.
H. W. HARMAN.



ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Chemical Method for the Detection of a Previously Frozen Condition in Fruit. W. M. Dehn and M. C. Taylor. (*J. Ind. and Eng. Chem.*, 1920, 12, 977-979).—Sugars exercise a protective action in fruit against freezing; cane sugar disappears in rotting fruit, and is transformed into invert sugar during the stages of ripening and over-ripening; cane and invert sugar are differently localised in the tissues of the fruit. The method described below makes use of the fact that freezing brings about a very rapid transformation of cane to invert sugar, resulting from the free mingling of cane sugar with inverting acids or invertase, or both. One half of the fruit to be examined is analysed directly, and the other half is frozen with ice and salt. Each sample of the fruit may be cut into halves, or, with very small fruit, two approximately equal samples may be set aside for the analysis. The directly analysed portion involves the estimation of invert sugar before and after hydrolysis; the frozen portion is also analysed for invert sugar before and after hydrolysis. In other words, the ratios of invert sugar to cane sugar before and after freezing are compared. In all cases the estimations are made by weighing the cuprous oxide formed after reduction. In the case of some peaches it was found that the freezing and thawing produced in fifteen hours three times as much inversion as storage for two weeks. By inspection of the values found for the percentage of inversion obtainable from the fruit before and after laboratory freezing, reliable conclusions may be drawn as to whether the fruit has already been subjected to cold storage or not, since the change brought about by freezing and storage will not be repeated by laboratory freezing, provided the first freezing was thorough and the laboratory freezing was not delayed too long. It will also be observed that not only cane sugar is inverted during rotting, over-ripening, rotting and freezing, but inverted sugar previously present or formed by inversion may be lost by chemical decomposition, or by metabolism of the plant, or by yeasts and moulds. The method takes advantage of divergencies in the largest components of the fruit, since inversion subtracts from the cane sugar concentration and adds to the invert sugar concentration.

H. F. E. H.

Detection of Incipient Putrefaction in Meat. R. Strohecker. (*Chem. Zeit.*, 1920, 44, 744).—Six 300 c.c. Winkler oxygen flasks are charged each with 5 grms. of sample and filled up with water at 40° C. They are incubated respectively for 0, 2, 4, 6, 8 and 10 hours at 23° C. Absence of oxygen after four to six hours, as shown by the Winkler method, indicates incipient putrefaction. Meat beginning to decompose (5 grms. in a 60 c.c. oxygen flask filled up with a nitrate solution containing 3 mgrms. N_2O_5 per litre) completely destroys the nitrate in two to four hours of incubation. Fresh meat requires much longer. Nitrite solution gives similar results. A method depending on the detection of amino acids by the "ninhydrin" reaction gave

less satisfactory results; methods based on the presence of other decomposition-products, as also on the concentration of hydrogen ions in fresh and decomposed meat, were unsuccessful. For the detection of advanced putrefaction 10 grms. of the sample, with 1 c.c. of Schardner's methylene-blue solution, are incubated at 45° C. in a 60 c.c. oxygen flask, completely filled with water and sealed. Distinct decolorisation in forty-five minutes indicates putrefaction. O. E. M.

New Dye for the Recognition of Abnormal Milk. I. J. O. de Vries. (*Nederl. Tijdschr. voor Melk-hygiene*, 1920, 83; through *Chem. Zeit.*, 1920, 44, *Rep.* 245.)—V. Slyke and Baker suggest the use of bromocresol purple (dibromo-o-cresol-sulphonphthalein) for the rapid detection of abnormal milk. The author has investigated their statements. On the addition of 1 drop ($\frac{1}{10}$ c.c.) of the saturated aqueous solution of the dye to 3 c.c. of the milk, a coloration is developed: greyish-blue with normal milk, dark blue with a lower content of acid, and light green to light yellow with a higher content. The higher content of acid may be due to formation or addition of acids, acid salts, or formaldehyde, or too great a degree of pasteurisation. If the colour is bluish, the milk is from an inflamed udder, or is watered or deprived of cream, or alkali has been added. O. E. M.

Concentration of Hydrogen Ions in Milk. J. Tillmans. (*Chem. Zeit.*, 1920, 44, 744.)—Although a normal raw milk may show increasing acidity by the Soxhlet and Hempel methods, its PH' remains constant. A normal milk will not pass the alcohol test if its PH' reaches 6 on the Sørensen scale. The alizarine test of Morres is nothing more than the alcohol test, with simultaneous estimation of PH'. It was shown that the acidity of fresh milk is due not to lactic acid, but to primary and secondary phosphates. The behaviour of boiled milk differs from that of fresh; either it undergoes a delayed lactic acid fermentation, or putrefies with production of alkaline bodies. Measurement of PH' was useless as a means of detecting neutralised milk, but specific conductivity determinations revealed addition of bicarbonate. Preservatives did not affect the PH', nor did removal of cream or addition of water up to 60 per cent. The reductase of Schardinger was the most sensitive to increase of PH', catalase the least, while the Storch enzyme occupied an intermediate position. Tubercular and other diseases were not revealed by abnormal figures, and milk from goats and sheep gave the same results as cows' milk. O. E. M.

Estimation of Moisture in Beet-Sugar Factory Products. V. L. Aikin. (*J. Ind. and Eng. Chem.*, 1920, 12, 979-981.)—A large number of results for moisture estimations of beet-sugar products are recorded, in which the method of mixing with sand is adopted. It is recommended that only sand be used which will pass a screen with 0.25 mm. perforations, the sand being previously digested in hot hydrochloric acid, washed and ignited. Twenty to twenty-five grms. of the sand are dried and weighed just previous to the estimation. Not more than 1 gm. of the material to be dried is then added, together with 1 c.c. of water; the dish is placed on top of the oven until warm, and the contents then mixed for three minutes until a perfectly homogeneous mixture is obtained. The whole is dried at a temperature of 105° C.

for six hours, and re-weighed until the loss in weight, after heating for a period of one hour, is less than 0.1 per cent. All weighings are made as soon as the temperature of the desiccator is within 2° C. of the temperature of the balance. Any determination which does not check within 0.2 per cent. should be repeated. H. F. E. H.

Chemical Composition of Natural Rice and of Italian Polished Rice.

G. Issoglio. (*Estratto dagli Atti della Reale Accademia delle Scienze di Torino*, 1919, 53, 731-744; through *Int. Rev. Sci. and Prac. of Agric.*, 1919, 10, 877-8.)—The author, with a view to contributing to the study of the most rational utilisation of cereals, has undertaken numerous investigations on the qualities of the following kinds of rice, specially with regard to their phosphatic constituents:

I. *Riso svestito rosso* (red hulled rice), composed of homogeneous grains covered with a red skin and perfectly white inside; the red colour is fairly resistant to acids, but is entirely removed by alkalis, specially ammonia. II. *Riso svestito bianco* (white hulled rice), which stands cooking well, giving out the characteristic smell of this cereal. III. *Riso brillato perla* (polished pearl rice), formed of small, transparent, heavy grains, slipping easily over each other; stands cooking well, but does not give off any agreeable odour. IV. *Riso mercantile raffinato* (refined commercial rice), white, homogeneous, stands cooking badly, gives off no agreeable odour, insipid. V. *Riso cremonese bianco* (white Cremona rice), similar to IV. VI. *Riso cremonese striato di rosso* (red striped Cremona rice), used with V. in experiments by Professor Guareschi, in the course of which pigeons and fowls preferred white Cremona rice. VII. *Riso bianco mercantile* (white commercial rice), kind placed on the market in 1918; has the organoleptic characters of milled rices with negative characters of appetibility.

The following table shows the results of the analysis of each of these 7 qualities:

	I	II.	III.	IV.	V.	VI.	VII.
Specific gravity ...	1.444	1.426	1.458	1.430	1.428	1.438	1.435
Water ...	Per Cent. 12.74	Per Cent. 13.22	Per Cent. 13.65	Per Cent. 13.28	Per Cent. 12.88	Per Cent. 12.65	Per Cent. 13.37
Ash ...	1.43	1.24	0.87	0.53	0.59	0.68	0.57
Fats ...	7.15	2.08	0.64	0.58	0.65	0.77	0.68
Nitrogen ...	1.53	1.37	1.25	1.27	1.30	1.51	1.28
Crude albuminoids ...	8.57	7.81	7.12	7.23	7.41	8.55	7.29
Cellulose ...	1.21	1.15	0.18	0.22	0.25	0.32	0.24
Pentosans ...	0.35	0.30	0.05	0.08	0.12	0.18	0.10
Starch ...	73.55	73.20	77.19	78.08	78.10	76.95	77.75
Total phosphoric anhydride P_2O_5 ...	0.84	0.68	0.26	0.24	0.38	0.46	0.29
Phytinic phosphoric acid ...	0.485	0.435	0.058	0.055	0.152	0.188	0.082
Lecithinic phosphoric acid ...	0.010	0.008	traces	traces	—	—	—
Phosphoric acid proportional to 100 parts of ash ...	58	55	30	45	62	67	50

The difference between hulled rice and milled rice is evident: the former is fairly rich in both mineral and organo-mineral phosphatic compounds, while the latter is poor in these constituents. Thus the amount of phosphoric pentoxide may be as high as 5 grms. per kgrm. in hulled rice, while it does not reach 1 grm. per kgrm. in refined polished rice; lecithinic phosphoric pentoxide is entirely absent from the milled rice, while in hulled rice, in the form of phospholecithin, it is as much as 0.1 grm. of lipid per kgrm.

It is also desirable to draw attention to the chemical affinity which exists between the phosphate derivatives and albuminous matter, from which comes the constant fact that rice rich in phosphates is also rich in albuminoids.

COMPARATIVE ANALYSIS OF 1 GRM. OF WHOLE RICE AND OF 1 GRM. OF POLISHED RICE.

					Whole Rice.	Polished Rice
					Per 1,000.	Per 1,000.
Phosphorus	...	...	...	...	2.678	0.890
Potassium	...	...	...	...	2.469	0.690
Magnesium	...	...	...	...	0.865	0.270
Sodium	...	...	...	...	0.434	0.210
Calcium	...	...	...	...	0.266	0.080
Iron	...	...	...	...	0.144	0.070
Chlorine	...	...	...	...	0.115	0.150
					6.871	

All this confirms the argument of Professor Guareschi that polishing decreases the valuable nutritive elements in rice.

Estimation of Sweetening Power. R. Pauli. (*Chem.-Zeit.*, 1920, **44**, 743.)

—This estimation has not hitherto been based on accurate methods, and the author outlines an improved experimental psychological procedure, including the interpretation of the results obtained. The method depends on the comparison by tasting of a number of pairs of solutions, one member of each pair being a sugar solution of unvarying concentration, the other member one of a series of saccharin solutions varying by equal stages within fixed limits above and below the sugar solution in sweetness. The conditions of test must be rigidly laid down and observed, and a number of observers is necessary to eliminate personal error. O. E. M.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Estimation of Aminoethanol and of Choline appearing in the Hydrolysis of Phosphatides. P. A. Levene and T. Ingvaldsen. (*J. Biol. Chem.*, 1920, **43**, 355.)—The following method is a modification of the one devised by Thierfelder and Schulze (*Zeitsch. physiol. Chem.*, 1916, **96**, 296). The ether-soluble acetone-insoluble egg lipoids are freed from white matter and hydrolysed by boiling with 3 per cent. sulphuric acid for eight hours. After filtration the sulphuric acid is precipitated by barium hydroxide, the filtrate concentrated in vacuo and basic lead acetate added

until no further precipitation occurs. The precipitate is filtered off, and excess of lead removed from the filtrate by sulphuretted hydrogen, when hydrochloric acid is added, and the solution evaporated with water repeatedly to remove acetic acid. The residue is then extracted with absolute alcohol, filtered, evaporated, and the residue of mixed bases dissolved in water and diluted to 25 c.c. Of this solution 15 c.c. are evaporated on a water-bath to a small volume, mixed with excess of calcium oxide, and extracted by boiling for a few minutes with three 75 c.c. portions of dry acetone. After filtration the combined extracts are acidified with hydrochloric acid and evaporated in vacuo, the residue being dissolved in water and evaporated to 10 c.c. Excess of strong hydrochloric acid is then added to 9 c.c. of the aqueous solution, followed by 1.9 grms. of gold chloride. On standing in a desiccator over sulphuric acid large crystals separate, which, after recrystallisation from dilute hydrochloric acid, melt at 190-192° C., and contain 49.11 per cent. of metallic gold, the theoretical amount for aminoethanol gold chloride being 49.17 per cent. The residue from the acetone extract is treated with water, filtered, the excess of hydrochloric acid and of calcium being removed by silver oxide and carbon dioxide respectively, treated with alcoholic picric acid solution, concentrated in vacuo, and allowed to cool in an ice chest. The precipitate of choline picrate is filtered off, and recrystallised from water, yielding long needles, having a melting point of 241-242° C., and containing 16.75 per cent. of nitrogen, comparing well with the theoretical value of 16.86 per cent.

T. J. W.

Estimation of Diastatic Power. E. I. Rosenblum. (*J. Soc. Chem. Ind.*, 1920, **39**, 311-313r.)—The difference in hydrogen ion concentration of starches due to the presence of acid salts renders the estimation of diastatic power untrustworthy. The diastatic power is at a maximum when the digesting solution has a faintly acid reaction towards methyl-red but is alkaline towards methyl-orange. This condition may be secured by the addition of 0.5 per cent. of ammonium dihydrogen phosphate to the solution. The diastatic power is estimated as described by Harrison and Gair (*Pharm. J.*, 1906, **94**, 6). Twenty c.c. of a solution, containing 0.1 gm. of malt extract, are added to a starch solution, containing 1 gm. of dry starch per 80 c.c. of water, which has been prepared by boiling and cooling to 46° C.; the mixture is kept at 46° C. for thirty minutes, a few drops of 20 per cent. sodium hydroxide solution are then added, the mixture diluted to 100 c.c., and titrated with Fehling's solution. This titration is termed T_D ; a further titration of a 1 per cent. malt extract alone gives T_M , a measure of the sugar originally contained in the extract. The formula $9531 \left(\frac{1}{T_D} - \frac{1}{T_M} \right)$ gives the diastatic power.

W. P. S.

Estimation of the Saccharogenic Power of Diastatic Preparations. K. Oshima. (*J. Ind. and Eng. Chem.*, 1920, **12**, 991-993.)—The author criticises adversely the Lintner method for the estimation of diastatic power, together with the Ling modification as adopted by the Institute of Brewing. He states that in the Ling modification there is no accurate agreement with the Lintner scale, since the quantity of enzymes and production of sugar do not give an exact proportion; par-

ticularly is there a noticeable difference when the production of sugar is too much or too little. To obviate these difficulties, Sherman and his associates proposed a method which depends upon the gravimetric estimation of copper oxide. The method is, however, for that reason too troublesome for routine factory work. The author then describes his own method as follows: In the case of a solid sample, it must be first extracted with distilled water for three hours at room temperature. From 1 to 10 grms. of the sample are usually extracted in 100 c.c. of water. If the enzyme is too concentrated, the solution is further diluted with a known amount of distilled water. Ten c.c. of the extract or the diluted extract are put into a flask containing 100 c.c. of 2 per cent. soluble starch solution, which has been placed in a water-bath kept at 40° C., the contents of flask are mixed, and the flask is allowed to remain in the bath for thirty minutes. At the end of that period, 10 c.c. of $\frac{N}{2}$ sodium hydroxide are added at once to stop further enzymatic action. Graduated quantities of the digest are then poured into a series of test tubes, each containing 5 c.c. of Fehling's solution; and the tubes are placed in a boiling water-bath for ten minutes, the tubes being shaken once during that period. The smallest quantity of the digested starch solution which just reduces 5 c.c. Fehling's solution is determined. It may be more convenient to use at first only two or three tubes containing quantities of the digest within wide regions of variations, so as to determine the approximate concentration of the digest to be used. The Lintner value according to tables and formulæ appended was then calculated from the amount of digested starch solution which will just reduce 5 c.c. of Fehling's solution. Experiments are described from which a culture of *Aspergillus oryzae* and an extract of malt were used, and it is claimed that the method is very convenient for experimental studies on various diastatic products, taka diastase, etc. It may also be used for the study of the influence of alkalinity, acidity, antiseptics, salts, etc. Results of experiments are also described in which a temperature of 50° C. is used for the enzyme action. H. F. E. H.

NOTE BY ABTRACTOR.—The above method appears to possess no advantages whatever over the Lintner "tube" method or the Ling modification thereof, nor does the author appear to be aware that the amount of maltose formed is only a linear function of the diastase, when means are taken to keep the starch conversion within the limits of Kjeldahl's law of proportionality. This is provided for both in the Lintner method and in Ling's modification.

Colorimetric Estimation of Histidine in Protein. M. T. Hanke and K. K. Koessler. (*J. Biol. Chem.*, 1920, **43**, 527.)—From 1 to 3 grms. of protein are mixed with 60 c.c. of 20 per cent. hydrochloric acid and hydrolysed by boiling for twenty-eight hours. Volatile matters are removed by distillation *in vacuo* at 60° C., and the residue finally dried *in vacuo* at 80° C. for one hour. The residue dissolved in 100 c.c. of water is treated with excess of calcium oxide and 50 c.c. of 90 per cent. alcohol, and then distilled *in vacuo* at 40° C. to remove ammonia, alcohol, etc. The residual solution is filtered and the precipitate washed with large volumes of hot water. To the alkaline filtrate is added a slight excess of hydrochloric acid, the liquid evaporated to dryness, dissolved in 18 c.c. of 37 per cent. hydrochloric acid,

diluted to 100 c.c., and heated on a water-bath; 100 c.c. of hot aqueous 15 per cent. phosphotungstic acid are added, and the mixture allowed to digest for thirty minutes. It is then left to cool slowly, and finally for three days in an ice-bath. The cold solution is filtered rapidly, the residue well washed with 200 c.c. of an ice-cold solution containing 3.3 grms. of hydrochloric acid, 15 grms. of phosphotungstic acid, and previously saturated with histidine phosphotungstate. The precipitate is dissolved in just sufficient 3*N* sodium hydroxide, filtered, and diluted to 1,000 c.c. This solution is used for the colorimetric estimation as previously described (*J. Biol. Chem.*, 1919, 39, 497).

Histamine and tyramine both yield a similar pink colour with *p*-phenyldiazonium-sulphonate, but are never present in sufficient quantity in non-putrid protein matter to affect the estimation of histidine.

Several tables are furnished giving results obtained by the above method with various proteins, and the effect of the presence of other amino-acids upon the estimation.

T. J. W.

Colorimetric Estimation of Histamine in Proteins. M. T. Hanke and K. K. Koessler. (*J. Biol. Chem.*, 1920, 43, 543.)—The dry solid is hydrolysed without preliminary treatment, but hydrated tissues are mixed with sufficient alcohol to give a final alcoholic strength of 75 per cent., a few drops of acetic acid added, and the mixture heated on a water-bath for two hours. After cooling and filtering, the residue is washed with 95 per cent. alcohol, and the alcohol evaporated from both residue and filtrate. The dry solid is hydrolysed, purified, and converted into histamine phosphotungstate by the methods described for histidine (see previous abstract). The phosphotungstate precipitate is suspended in a large volume of hot water, treated with excess of saturated barium hydroxide solution, and digested for one hour on a water-bath. After cooling, the solution is filtered, the filtrate freed from excess of barium by the careful addition of $\frac{1}{2}$ sulphuric acid, the mixture filtered hot, and evaporated to dryness. From the residue, by a complicated series of reactions and colorimetric determinations, for which the original paper must be consulted, the percentage of histamine is determined. Experiments are described, showing that histamine is quantitatively precipitated by phosphotungstic acid, by silver nitrate and by baryta, and is extracted to the extent of 96 per cent. by a mixture of chloroform and methyl alcohol.

T. J. W.

Adamkiewicz Protein Reaction and a New Colour Test for Glyoxylic Acid. W. R. Fearon. (*Biochem. J.*, 1920, 14, 548.)—The work described in this paper was designed to find a satisfactory method for the colorimetric estimation of tryptophane, and the nature of the aldehydes concerned in the Hopkins-Cole and Rosenheim reactions in order to decide the glyoxylic acid-formaldehyde controversy.

The reagent used for the glyoxylic acid test is a 1 per cent. solution of pyrogallol in pure concentrated sulphuric acid free from nitrous contamination. This will keep in satisfactory condition for a few days only. On adding a trace of glyoxylic acid and gently warming, a deep blue colour gradually develops, which

becomes carmine on dilution with water, and again blue on the addition of sulphuric acid in excess. With the aliphatic aldehydes the reagent gives various shades of red.

The author concludes as a result of this work that the Hopkins-Cole test is due to glyoxylic acid, while the Rosenheim reaction depends upon the presence of formaldehyde.

The following qualitative test for tryptophane and the indoles is given: the reagent is a 10 per cent. solution of salicyl aldehyde in alcohol free from acetone. To the liquid under examination is added excess of strong hydrochloric acid, 4 or 5 drops of the reagent, and the tube is gently warmed for a few minutes. One drop of 10 per cent. hydrogen peroxide is then added and the warming continued, when tryptophane yields an intense blue colour, skatole gives a deep purple, while indole produces a bright carmine. Care must be taken not to add excess of hydrogen peroxide or the colours will be destroyed. T. J. W.

Proximate Analysis of Hardwoods: Studies on *Quercus Agrifolia*.

W. H. Dore. (*J. Ind. and Eng. Chem.*, 1920, **12**, 984-987.)—In a previous paper (*ANALYST*, 1919, **44**, 299) the author has described methods for the analysis of wood, and given the results of three coniferous woods and two hardwoods. It was then shown that the procedure succeeded with coniferous woods, but not with hardwoods, owing to the fact that König's 72 per cent. sulphuric acid method for lignin does not give reliable results for the woods of the broad-leaved trees. In the present paper the author employs the gaseous hydrochloric acid method of König and Becker (*J. Soc. Chem. Ind.*, 1919, **38**, 530A). This method is described in detail, for which the original paper should be consulted. The composition of oak wood was found to be that given in the following table, the figures in all cases being the average of two or more determinations.

COMPOSITION OF LIVE OAK WOOD (*Quercus Agrifolia*).

Constituent.						Air-dry Basis. Per Cent.
Loss on drying	...	...	...	...	...	4.20
Benzene extract	...	...	...	...	...	0.50
Alcohol extract	...	...	...	...	...	4.33
Soluble in cold water	...	...	...	...	...	3.66
Soluble in cold 5 per cent. sodium hydroxide	...	...	...	...	...	18.71
Cellulose	...	...	...	...	...	45.48
Lignin	...	...	...	...	...	20.25
Pentosans not otherwise accounted for	...	...	...	...	...	1.89
Mannan (residual)	...	...	...	...	...	—
Galactan (residual)	...	...	...	...	...	1.49
Total	...	...	...	...	...	100.51

In this table the first five groups of substances represent extraneous matter removed from the wood in the course of the purification process, while the last five groups represent substances contained in the residual purified tissue. The amounts obtained for each of the first five groups vary considerably with the manner in which the determinations are carried out, but the weight of purified residue remaining is in all

cases about the same. The purification treatment employed for the tissue of coniferous woods (successive extraction with benzene and alcohol) was supplemented with digestion in cold water and cold 5 per cent. sodium hydroxide solution. These treatments remove all adventitious substances, but do not injure either the cellulose or the lignin. The modified procedure conforms to the requirements of a satisfactory analytical scheme, in that it accounts for all of the material of the wood, yields the maximum amount of main constituents free from impurities or degradation products, avoids overlapping of constituents, and overlooks no constituents of importance.

H. F. E. H.

ORGANIC ANALYSIS.

Estimation of in Cineol Essential Oils. C. Kleber and W. F. von Rechenberg. (*J. prak. Chem.*, 1920, 101, 171-176.)—A method which may be used for the estimation of cineol in eucalyptus oil depends on the determination of the solidifying point of the oil. Pure cineol solidifies at 1.2° C. The following table gives the solidifying points of eucalyptus oil containing various amounts of cineol :

Cineol, per Cent.	Solidifying Point.	Cineol, per Cent.	Solidifying Point.
100	1.2°	82	- 8.0°
98	0.3°	80	- 9.2°
96	- 0.7°	78	- 10.3°
94	- 1.7°	76	- 11.5°
92	- 2.7°	74	- 12.8°
90	- 3.8°	72	- 14.1°
88	- 4.8°	70	- 15.4°
86	- 5.8°	68	- 16.7°
84	- 6.9°		

Normal eucalyptus oils do not contain less than 70 per cent. of cineol; in the case of oils containing less than this quantity it is advisable to add 50 per cent. of pure cineol before determining the solidifying point, and to make a correction subsequently for the quantity added.

W. P. S.

Estimation of Creatinine. E. Vautier. (*Ann. Chim. anal.*, 1920, 2, 300-305.)—The following modification of the picric acid method is suitable for the estimation of creatinine in "concentrated bouillon" and similar preparations. From 0.25 to 3 grms. of the substance, according to the probable amount of meat extract present, is evaporated with hydrochloric acid, and the residue taken up with boiling water, and filtered through a moistened filter to remove fat, etc. The filtrate is again evaporated to dryness on the water-bath, with the addition of a few drops of concentrated hydrochloric acid, and the mass frequently stirred to prevent the crystals of sodium chloride which separate enveloping some of the liquid containing creatinine. The dry or nearly dry residue is cooled, treated with 10 c.c. of water, and then with 15 c.c. of picric acid solution (1.2 per cent.) and 8 c.c. of 10 per cent.

sodium hydroxide solution, shaken, and, after five minutes, made up to 500 c.c., and the creatinine estimated colorimetrically. For this purpose the colour may be compared with that of a $\frac{N}{2}$ solution of potassium dichromate in a layer of 8 cm. in depth. This coloration is matched approximately by the following amounts of creatinine :

Creatinine, mgrms. ...	2.5	3.0	3.5	4.0	4.5	5.0	5.5	6.0	6.5	7.0	8.0	9.0	10.0	12.0	15.0	20.0
Depth of liquid, cms.	18.0	14.5	11.5	9.9	9.5	8.6	7.7	7.2	6.7	6.3	5.5	4.8	4.2	3.6	2.8	2.2

The intermediate values may be obtained by interpolation.

C. A. M.

Microchemical Identification of Cyanic Acid. R. Fosse. (*Comptes rend.*, 1920, 171, 635-637; 722-723.)—Cyanates, when heated with water and ammonium chloride, yield urea, which may be identified by means of xanthhydrol. Characteristic crystals are obtained when 0.005 gm. of silver cyanate is boiled with 5 c.c. of water and the solution then cooled. A blue coloration is obtained when 1 mgrm. each of silver cyanate, potassium chloride, and crystallised cobalt acetate are ground together in a porcelain basin; the colour is destroyed by water, but re-appears when alcohol is added. Cobalt thiocyanate has also a blue colour, but it is insoluble in amyl alcohol, is not decolourised by dilute acids, and gives a red coloration with ferrie salts, and may thus be distinguished from the blue potassium cobalticyanate.

W. P. S.

Sensitive Form of the Lieben Iodoform Reaction. R. Kunz. (*Zeitsch. anal. Chem.*, 1920, 59, 302-303.)—The test consists in warming 10 c.c. of the solution between 50°-60° C. with 1.5 to 2 c.c. of 1 in 10 sodium hydroxide solution, about 0.15 gm. of potassium iodide, and 0.2 gm. potassium persulphate. A solution containing 1 drop of alcohol in 100 c.c. water gives a turbidity after ten minutes.

O. E. M.

Distillation of Petroleum, Petroleum Residues, or other Oils immiscible with Water. H. Offermann. (*Chem. Zeit.*, 1920, 44, 773-774.)—Wet steam is used at ordinary pressure instead of the superheated steam, with or without reduction of pressure, prescribed by Engler. Cracking is thus avoided, and laboratory and refinery results are brought to the same level. Steam from a conical flask is conveyed upwards, with partial condensation, to a dip tube passing through the tubulure to the bottom of a weighed retort, the neck of which rises to an adapter attached to a Liebig condenser. The charge of oil, 500-1,000 grms., having been heated to boiling, the steam is passed through at such a rate that at the beginning the distillate consists of 25 per cent. water, and 75 per cent. oil, in the middle 50 per cent. of each, and towards the end 75 per cent. water and 25 per cent. oil. Comparative results from a petroleum residue show that lower sp. gr. and flash-point of the fractions produced, a higher proportion of pitch and loss, and production of pitch of higher melting-point and greater insolubility in benzene, accompany the use of steam superheated to 250-400° C.

O. E. M.

INORGANIC ANALYSIS.

Estimation of Carbon Dioxide in Alkali Bicarbonate containing Carbonate. W. Hartmann. (*Zeitsch. anal. Chem.*, 1920, 59, 289-297.)—The substance equivalent to 0.1-0.3 grm. bicarbonate, together with small pieces of pumice and short lengths of 2 mm. bore glass tubing, is placed in a boiling-tube, 15 × 3 cm., closed by a 4-hole rubber stopper. This carries a 200° C. thermometer and an inlet tube, both reaching the bottom, and also a tap-funnel and an outlet tube. Air freed from carbon dioxide by bubbling it through potash solution is drawn through the apparatus, and thence in succession through cooling bulbs, strong sulphuric acid in a washing bottle surmounted by a water-jacketed tube, a calcium chloride tube, a weighed potash absorption apparatus, and a calcium chloride tube, to a laboratory pump. After admission through the tap funnel of 7 c.c. of 70 per cent. glycerol for each 0.1 grm. of carbonate taken, the contents of the tube are heated for ten minutes at 117° C. for sodium bicarbonate or 114° C. for potassium bicarbonate. Air is now drawn through for thirty minutes at 90-100° C., and the potash bulb weighed. For the normal carbonate the temperature is raised to 180°-190° C. for five minutes, 2 drops of water are added, and heating continued for five minutes. Air is drawn through for thirty minutes at 150° C. before weighing. Any ammonia present can be estimated by rendering the sulphuric acid alkaline and distilling. Anhydrous normal carbonate, especially sodium carbonate, requires heating at 180-190° C. for forty minutes, with addition of 2 drops of water every two minutes. The table of results shows that although normal carbonate, especially potassium carbonate, gives off considerable quantities of carbon dioxide at 115-120° C., the bicarbonates at these temperatures give results agreeing with theory. With bicarbonates, high and low results respectively are produced by the admixture of salts which promote dissociation, and an excess of normal carbonate. The method is only applicable to bicarbonates alone or in admixture with non-electrolytes.

O. E. M.

Determination of Carbon Monoxide in Air contaminated with Motor Exhaust Gas. M. C. Teague. (*J. Ind. and Eng. Chem.*, 1920, 12, 964-968.)—Motor exhaust gas is found to contain the following constituents: carbon dioxide, carbon monoxide, oxygen, hydrogen, methane, traces of unsaturated hydrocarbons, and some unburned gasoline; there are also present water and varying amounts of oil-smoke particles. The iodine pentoxide method for determining low concentrations of carbon monoxide was investigated with reference to its use with dilute motor exhaust gas. The form of apparatus hitherto used was found to give appreciably higher results, owing to the presence of small amounts of unburned gasoline. A new apparatus was therefore devised, and is fully described and illustrated. All the interfering gases are first removed at the temperature of liquid air (−190° C.); at this temperature liquid gasoline becomes a glass-like solid, while all the water, carbon dioxide, and probably all the unburned hydrocarbons are also condensed. The method is found quite satisfactory for determining carbon monoxide in small quantities in

the presence of gasoline vapour. A portable apparatus is also described and illustrated, which permits a determination to be made in fifteen minutes with an accuracy of 0.003 to 0.005 per cent. carbon monoxide; this may be of use in exploring vehicular tunnels, garages, and other enclosed spaces. H. F. E. H.

Separation of Iron from Aluminium by Precipitation as Prussian Blue. H. Hale and G. O. Burr. (*J. Amer. Chem. Soc.*, 1920, **42**, 2056-2058.)—The quantitative separation of iron from aluminium by precipitation as Prussian blue is not practicable, owing to the aluminium interacting with the excess of ferrocyanide to form a jelly which cannot be manipulated. The addition of acid retards, but does not prevent the formation of the jelly. Coagulation of the Prussian blue is effected by the addition of various salts, especially aluminium chloride; but, even after coagulation, quantitative filtration is impossible, although good results were obtained by centrifuging. C. A. M.

Volumetric Estimation of Mercury, Iron, and Vanadium in the Same Solution. G. Hinard. (*Ann. Chim. anal.*, 1920, **2**, 297-299.)—I. *In the Highest Stage of Valency.*—The solution of the three metals in dilute sulphuric acid is treated with hydrogen sulphide, the mercuric sulphide separated and oxidised with bromine-water, the mercury precipitated as hydroxide, which is dissolved, without filtration, in potassium cyanide solution, and the excess of cyanide titrated with standard silver nitrate. The filtrate from the mercuric sulphide is boiled, and oxidised with bromine-water, the iron precipitated with potassium hydroxide, re-dissolved in hydrochloric acid, and reprecipitated. It is then dissolved in warm sulphuric acid, the solution cooled, treated with excess of potassium iodide, and the iron estimated by titrating the liberated iodine with standard thiosulphate solution. Another portion of the original solution is rendered just alkaline, the incipient precipitate redissolved in a slight excess of sulphuric acid, and the iron and vanadium estimated together iodimetrically, the titration with thiosulphate being continued in this case until the liquid becomes blue. The vanadium is then found by difference. II. *In Different Stages of Valency.*—The total quantities of the three metals are estimated as described above. Another portion of the original solution is treated with sodium chloride, which precipitates mercurous salts, and the mercuric salts are then precipitated with hydrogen sulphide from the filtrate. *Iron and Vanadium.*—(a) *Absence of ferrous salts:* Vanadic acid is estimated iodimetrically as described, and a deduction made for the amount of thiosulphate corresponding to the iron already estimated. The vanadium, present as a vanadyl salt, is obtained by difference. (b) *Presence of ferrous salts, absence of vanadates:* The solution is treated, without prior oxidation with bromine-water, with potassium iodide. In this case the liberated iodine corresponds to the ferric iron. The ferrous iron is found by difference, whilst the total vanadium has been already determined. C. A. M.

Determination of Nitrates in Soil. A. L. Whiting, T. E. Richmond, and W. R. Schoonover. (*J. Ind. and Eng. Chem.*, 1920, **12**, 982-984.)—The method

suggests modifications in the Devarda method, and depends upon the oxidising action of sodium peroxide and the completeness of extraction of the nitrate with hydrochloric acid. The water-free samples from moisture determinations are placed in 400 c.c. shaker bottles, and 300 c.c. of approximately 0.5 per cent. hydrochloric acid are added. The mixture is then shaken in a mechanical shaker for one to three hours, and allowed to settle overnight. Five grms. of sodium peroxide are placed in a 800 c.c. Kjeldahl flask. A 200 c.c. aliquot part of the acid soil extract is blown off into a graduated flask, then poured on the peroxide, and the contents of the flask boiled down to 20 to 25 c.c., or, if urea is present, to complete dryness. Two hundred c.c. of nitrogen-free distilled water are now added, together with 0.5 gr. of Devarda's alloy (50 per cent. aluminium, 45 per cent. copper, and 5 per cent. zinc), and the mixture distilled for forty minutes, and collected in standard sulphuric acid. Sodium hydroxide of a strength such that 1 c.c. = 0.5 mg. of nitrogen is used for titration. Rosolic acid or methyl red is the best indicator. Hydrochloric acid was selected as the best acid for extraction, a solution of 0.2 per cent. strength being very satisfactory where soils are not heavily treated with carbonates. Between the limits 0.2 and 1 per cent. the strength of the acid was found not to affect results. By the use of sodium peroxide an alkaline solution is left which expels the ammonia and leaves the proper conditions for reduction with Devarda's alloy. The method determines only nitrate nitrogen, as confirmed by precipitation of the nitrate in the soil extracts with 10 per cent. nitron solution in 5 per cent. acetic acid, followed by an analysis of the crystals of nitron nitrate and the filtrate from the same. Slightly lower results are given by the Devarda method, when modified as described, than by the nitron method. The method has been successfully used on thousands of soil samples, and also been applied with satisfactory results to crop samples.

H. F. E. H.

Examination of the Methods used in Estimating Phosphorus in Iron and Steel, Ores and Slags. H. Kinder. (*Chem. Zeit.*, 1920, **44**, 692-693.)—The magnesia method is slow, and for small quantities of phosphorus, inaccurate. The molybdate method was examined, and the results of Finkener, who dries the precipitate at 105° C., and of Meinecke, who ignites to about 450° C., were confirmed. The presence of 1 per cent. of copper, 20 of nickel, or 5 of cobalt, is without effect, while 5 per cent. of chromium was doubtful. Tungsten takes down phosphorus, and the precipitate may be dried, dissolved in hydrochloric acid, oxidised with potassium chlorate, and the phosphorus determined as usual in the diluted filtrate. Molybdenum precipitates must be examined for phosphorus. Solutions containing vanadium should be reduced with ferrous sulphate or sodium sulphite before precipitation. Titanium causes the precipitation of some phosphorus; the precipitate is fused with soda, and phosphorus estimated in the extract. Arsenic is partially precipitated as molybdate, unless the concentration of the nitric acid or of the solution is increased, or the precipitation carried out below 65° C. Reduction and volatilisation of arsenious acid by means of hydrobromic acid is the best method. For ores or slags the weight taken for analysis should be 5 or 10 grms. for 0.1 per cent. phosphorus, down to 0.3 or 0.5 grm. for 2 per cent. The liquid for precipitation should not exceed 150 c.c., and

contain not over 5 c.c. strong nitric acid. The temperature should be 60-70° C., and 50-60 c.c. molybdate solution should be used. Settling, hastened by shaking, must be complete before filtering. The reagents should be measured, and a control performed with them in glass vessels similar to those used in the analysis, while fusion of residues must not be carried out in platinum crucibles used for igniting magnesium pyrophosphate. For iron and steel, volumetric estimation is necessary in the works on account of its quickness, and is sufficiently accurate if based on gravimetric estimations of samples similar to those under test. O. E. M.

Estimation of Potassium as Perchlorate. G. P. Baxter and F. E. Rupert. (*J. Amer. Chem. Soc.*, 1920, **42**, 2046-2048.)—Experiments described in detail show that in Baxter and Kobayashi's method of estimating potassium as perchlorate (ANALYST, 1920, 238), ethyl alcohol, denatured with 5 per cent. of methyl alcohol, may be used in place of absolute ethyl alcohol as the washing liquid, whilst the temperature is unimportant, if the washing liquid is saturated with the potassium perchlorate after the initial extraction, and the following modification of the method is used: The solution of the mixed potassium and sodium perchlorates is evaporated to dryness with a small quantity of perchloric acid, and the residue extracted with 20 c.c. of denatured alcohol containing 0.2 per cent. of perchloric acid, and washed with two portions of 5 c.c. of the same solution. The first extract and washings are filtered through a tared platinum sponge crucible. The potassium chlorate is then dissolved in water, the solution evaporated to dryness with a small quantity of perchloric acid, and the residue transferred to the platinum sponge crucible by means of denatured alcohol which contains 0.2 per cent. of perchloric acid and is saturated with potassium perchlorate. This solution is made by dissolving the necessary quantity of potassium perchlorate in warm concentrated perchloric acid, and adding the solution to the alcohol. From 75 to 95 c.c. are used for washing the residue in the crucible at a temperature up to 20° C. C. A. M.

Volumetric Estimation of Potassium. G. Ajin. (*Chem. Trade J.*, 1920, **67**, 501.)—To 25 c.c. of a 2 per cent. solution of potassium sulphate or chloride, 50 c.c. of 2*N* tartaric acid are added slowly, followed by 25 c.c. of $\frac{N}{2}$ aqueous sodium hydroxide, the flask being well shaken during the addition and for five minutes after. Twenty-five c.c. of 96 per cent. alcohol are run in very slowly with constant agitation, and the whole allowed to stand eight hours. The precipitate is filtered off, washed nine times with neutral 96 per cent. alcohol, transferred to a flask and exactly neutralised to phenolphthalein by the addition of $\frac{N}{10}$ sodium hydroxide in 96 per cent. alcohol. A known volume of $\frac{N}{10}$ aqueous sodium hydroxide is added until the precipitate is completely dissolved and the excess of the sodium hydroxide titrated with $\frac{N}{10}$ hydrochloric acid. The volume in c.c. of $\frac{N}{10}$ sodium hydroxide used in dissolving the precipitate, multiplied by 0.00471, gives the quantity of potassium oxide (K_2O) present. T. J. W.

Differentiation of Radium from Barium by Means of Iodic Acid. G. Denigès. (*Comptes rend.*, 1920, **171**, 633-635.)—The crystals obtained when a drop of barium bromide solution is treated with a drop of iodic acid solution are quite

different from those obtained under similar conditions from radium bromide solution. The difference is particularly marked when the concentration of the bromide solutions does not exceed 0.03 per cent. W. P. S.

Volumetric Methods for Estimating Tin. J. G. F. Druce. (*Chem. News*, 1920, 44, 173.)—The known methods were investigated. Titration of stannous salts in hydrochloric acid with standard iodine solution gave results agreeing well with the theory, and can be used for the inorganic stannochlorides and those of the aliphatic amines; most of the aromatic amines darken and obscure the end-point. Permanganate is satisfactory for use on fresh stannous solutions in sulphuric acid; a small quantity of sodium carbonate is added to prevent oxidation. O. E. M.

APPARATUS, ETC.

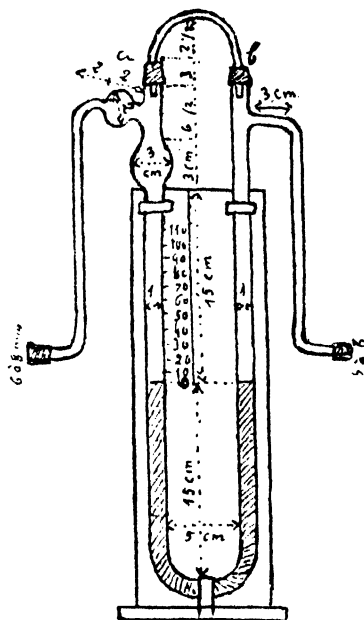
New Method of Measuring the Velocity of a Current of Gas. J. Erlich. (*Ann. Chim. anal.*, 1920, 2, 289-297.)—When a liquid flows under a pressure h , through a capillary tube, its velocity, v , may be represented by Poiseuille's formula—

$V = K h \frac{d^4}{l}$, where d represents the diameter of the

tube, l its length, and k a constant depending upon the nature of the glass of the tube and of the liquid. This law also extends to gases, but above a certain velocity (70 to 100 litres per hour) h increases more rapidly than the law indicates. When a gas escapes from a capillary tube at a velocity, v , it causes a difference of pressure h , apart from the tube. Since k is constant for a given gas and apparatus each value for v corresponds with a value for h which is always the same. An apparatus embodying this principle is shown in the accompanying diagram. A capillary tube ab is connected at its extremities with the limbs of a differential manometer m , the bulbs on which are intended to prevent the liquid reaching the capillary tube. The deviations of the level in the manometer are measured upon a scale graduated in tenths of a cm. In practice it is convenient to construct a graph for a given apparatus and gas, showing directly the values of v corresponding with the deviations in level h . It is essential that the gas should be dry. Changes of temperature are corrected by means of the formula—

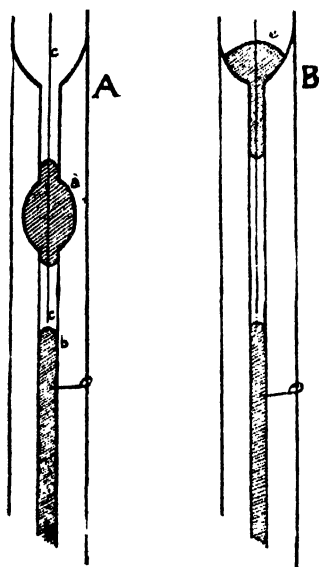
$$V_r = \frac{V}{1 + k(t - t^0)} \left(\frac{273 + t^0}{273 + t} \right)^3,$$

where t represents the temperature at which the gas was measured— t^0 the temperature of the determination, V the velocity read on the graph, V_r the true velocity, and k the coefficient of dilatation of the manometer liquid. C. A. M.



Laboratory Apparatus for Preparing Electrolytic Hydrogen. W. G. Palmer. (*J. Soc. Chem. Ind.*, 1920, 39, 316r.)—An unglazed earthenware pot, 3 by 7 inches, has three holes drilled in its closed end; vertical rows of small holes are also drilled in the sides, and the pot is soaked in melted paraffin wax. A large sheet of parchment paper is wrapped round the pot so as to cover the rows of holes and is held in position with rubber bands. A cylindrical cathode of sheet nickel is fixed in the pot, the leads passing through glass tubes cemented in two of the holes in the closed end, and the pot is then placed in an inverted position in a vessel containing 10 per cent. potassium hydroxide solution. A cylindrical anode of sheet nickel, previously immersed in concentrated nitric acid for thirty minutes is fitted in the vessel containing the electrolyte, a distance of about 0.3 inch being left between the anode and the parchment paper cover of the pot. The third hole in the end of the pot is fitted with a rubber bung and a delivery tube for the hydrogen. A current of about 10 ampères may be used.

W. P. S.



Thermo-Regulator. J. F. King. (*J. Amer. Chem. Soc.*, 1920, 42, 2058.)—A simple device is shown to prevent the oxidation of mercury in a thermo-regulator owing to the formation of the small arc when the contact is broken between the mercury column and the adjustable wire contact. In the accompanying diagram A represents the capillary of a regulator with a small bulb at *a*. A capillary pipette is passed down to *b*, and a stream of an inert gas is introduced, to displace the air in the capillary, which is then sealed with a few drops of mercury at *a*. This mercury rises and falls with the movement of *b*, but never falls down on to *b*. Any arc then formed between the wire and the mercury column at *b* will then be in an inert gas. In the case of an ordinary thermo-regulator, B, the same result may be attained by sealing the capillary with a drop of mercury at *c*.

C. A. M.

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GOVERNMENT REPORT.

Report of the Government Chemist upon the Work of the Government Laboratory for the Year ended March 31, 1920, with Appendices. Cnd. 881. (Price 3d. net. H.M. Stationery Office.)—The total number of samples examined during the year was 368,898, an increase of nearly 80,000 over the number for the previous year. The principal increases were due to goods liable to Customs duty, such as wines, sugar, tea, and cocoa preparations, and are indicative of the revival of trade; at the same time, there was naturally a considerable decrease in the number of samples submitted by such Departments as the Admiralty, Air Ministry,

and the War Department. Of the above total number of samples, 169,510 were examined at the outpost chemical stations.

Customs and Excise : Beer.—The number of samples examined in connection with the duty on beer was 25,489. *Materials used in Brewing :* The samples of malt, corn, brewing sugars, and exhausted grains numbered 323, and the yeast foods and miscellaneous samples 72. *Worts or Beer in the Unfinished Condition :* As a check on the assessment of beer duty 8,890 samples drawn from fermenting vessels were analysed ; 770 samples of beer, submitted as having become sour and unsaleable, were examined, and in most cases the beer was destroyed and the duty remitted or refunded. No evidence of dilution was found in samples of beer taken from the premises of publicans and other retailers. *Non-alcoholic Beers, Herb Beers, and Beer Substitutes :* Of 212 samples analysed, 81 were submitted as non-alcoholic beers, and 22 of these contained more than 2 per cent. of proof spirit. In one case the proof spirit exceeded 5 per cent. Of the remaining 132 samples (herb beers, ginger beers, etc.), 34 contained between 2 and 5 per cent. of proof spirit and 1 sample contained as much as 9·6 per cent. The importation of beer is still insignificant as compared with pre-war times, only 133 samples being examined. *Examination of Beer and Brewing Materials for Arsenic :* The number of samples tested, including beers, worts, malt, sugar, etc., was 969 ; of these, 5 were found to contain arsenic in excess of the limits suggested by the Royal Commission on Arsenical Poisoning. None of the samples of malt and sugar contained arsenic in excess of the limit, but the limit was exceeded in three instances out of 619 samples of beer and wort.

Cider and Perry.—Forty-four samples of imported cider were examined, of which 20 were classed as spirit preparations. Seven samples submitted by Customs and Excise officers were found to be factitious ; 7 samples of sour ciders, submitted for the purpose of obtaining remission of duty, were found to contain sufficient acid to render them unsaleable.

Table Water Duty.—Of 20 samples of mineral waters analysed, 16 proved to be medicinal and 4 were liable to table water duty ; of 333 cordials, syrups, etc., only 10 contained more than 2 per cent. of proof spirit, the highest amount being 11·3 per cent., and none of the 102 samples of so-called non-alcoholic wines contained more than 2 per cent. of proof spirit.

Spirits : Fusel Oil.—Only 1 out of 19 samples of fusel oil from distilleries was found to contain more than the permitted proportion of proof spirit. *Illicitly Distilled Spirits :* The analytical results afforded strong evidence that several of the 16 samples analysed had been distilled illicitly. *Duty-free Spirits, etc. :* Only 16 out of 848 samples of wood and mineral naphtha submitted were found to be unfit for methylating purposes. For the purpose of controlling the use of duty-free methylated and other spirit in connection with manufacturing processes, 539 samples of recovered spirit, etc., were examined. There was a considerable increase in the number of applications in respect of claims for rebate on alcohol used in making medicinal preparations, and for permission to receive methylated spirit and other forms of duty-free alcohol for manufacturing purposes and research.

Wines.—The total number of samples examined in connection with assessment of Wine Duty was 143,544, an increase of over 45 per cent. on the number for 1914.

Sugar, Glucose, and Saccharin.—The number of samples tested was 36,411 of sugar, 1,488 of glucose, and 321 of saccharin. In addition, 121 samples of imported substances likely to contain saccharin were analysed.

Tobacco.—During the year the percentage of moisture was determined in 10,234 samples, and the percentage of oil in 1,140 samples. The other samples examined comprised tobacco for drawback, 13,797; tobacco stalks, 18,893; offal snuff, 12,020; unmanufactured tobacco, 765; miscellaneous samples, 943.

In addition to the above the following number of samples were examined for this Department: Tea, 16,562; coffee and coffee substitutes, 1,527; cocoa and chocolate, 8,922; matches, 72 (no evidence of the presence of white phosphorus was discovered); medicines (under medicine stamp duty), 62; imports by parcel post, 590; etc.

Air Ministry.—Four hundred and ninety-five samples, mainly alloys, were examined for the Department, and special investigations were made on certain deposits found in the cylinders and piston heads of aircraft engines.

Ministry of Agriculture and Fisheries.—The samples examined for the ministry of Agriculture and Fisheries, the Board of Agriculture for Scotland, and the Department of Agriculture and Technical Instruction for Ireland numbered 1,023. *Imported Milk and Cream:* Five samples of fresh milk and 2 of sterilised milk were analysed; all were free from preservatives, but one sample of sterilised milk was deficient in fat. Three hundred and forty samples of condensed milk and dried milk were tested; the percentage of fat in the samples of whole condensed milk varied from 6·4 to 12·2 per cent. All the samples were free from preservatives and there was no evidence of skimming. Eight samples of cream contained from 8 to 10 per cent. of fat; preservatives were absent. *Imported Butter:* Only six samples were received; they were free from foreign fats, but 2 contained more than 16 per cent. of water. Of 267 samples of imported margarine, 5 contained more than 16 per cent. of water. *Imported Cheese:* The 37 samples of cheese analysed were all genuine. This ministry also submitted samples of sheep dips, rat poisons, oysters, fertilisers, insecticides, etc. *River Pollution:* In addition to the examination of 98 samples of river water and effluents, work was carried out in order to determine the nature and proportions of the toxic constituents which might be present in drainage from tarred roads.

Ministry of Munitions.—Amongst the samples examined for this Department were liquid fuel taken from a German tractor; salts and brines for the occurrence of potash and bromine; materials used in the recovery of potash from felspar, and dust in cement works flues. This dust was found to be rich in potash in a form readily available for use. Arrangements were made to extract radium from many thousands of luminous dials, gun sights, compass cards, etc., and practically all the radium present was recovered in a usable form and at comparatively small cost.

Ministry of Food.—During the year 3,073 samples were submitted; these included milk (fresh and condensed), jam, cheese, feeding stuffs, crude and refined oils, and edible fats. Many of the samples were taken in connection with the weekly supervision of factories under the control of the Ministry.

Ministry of Health.—Thirty-six samples of canned meat, condensed milk, and dried egg were examined.

A description is also given of the work done for the Crown Agents for the Colonies, Geological Survey, Home Office, India Office, Inland Revenue, Post Office, Stationery Office, Board of Trade, Corporation of Trinity House, War Department, Office of Woods, Office of Works, London, and Office of Works, Dublin.

Sale of Food and Drugs Acts.—The samples referred by magistrates under the Sale of Food and Drugs Acts of 1875 and 1899, and under Food Control Orders, numbered 121; these included 98 samples of milk, 8 of beer, 5 of whiskey, 3 of margarine, 2 each of butter, vinegar, and cream of tartar, and 1 each of ginger, gin, mustard, canned peas, camphorated oil, and sodium bicarbonate. Four of the samples could not be examined owing to their condition on receipt. The following are the particulars of the most important cases:—*Butter*: One sample contained foreign fat, and 1 excess of water, as alleged by the public analyst. *Vinegar*: Both samples consisted of artificial vinegar, as alleged. *Cream of Tartar*: The results agreed with those of the public analyst, who had found an excessive quantity of lead in the samples. *Beer*: The samples were referred in connection with proceedings under the Beer (Prices and Description) Order, 1919. In 1 case the original gravity differed from that found by the public analyst; in the remaining 7 cases there was no information as to the finding of the public analyst. *Whiskey and Gin*: The point in dispute for all the samples was the alcoholic strength, but it is not known whether or not the results were in agreement with those of the public analyst. In the case of the ginger alleged to contain spent ginger, the results did not agree with those of the public analyst. In the case of canned peas alleged to contain copper, camphorated oil alleged to be deficient in camphor, mustard alleged to contain foreign starch, and sodium bicarbonate alleged to contain arsenic, the results agreed with those of the public analyst. In the 89 cases of milk samples in which certificates were issued the charges were as follows: In 26 samples, added water; 45, abstraction of fat; 6, added water and abstraction of fat; 2, presence of preservatives; and 10, presence of dirt. In 7 cases the results were not in agreement with those of the public analyst.

Fertilisers and Feeding Stuffs Act.—The samples under this Act were submitted by the Ministry of Agriculture and Fisheries in connection with application by local authorities for the consent of the Board to take proceedings against the sellers. The 13 samples of fertilisers received consisted of basic slag, hair and fur waste, guano, and plant foods; the 5 samples of feeding-stuffs consisted of feeding meals and poultry foods.

W. P. S.



REVIEW.

THE CARBOHYDRATES AND ALCOHOL. By S. RIDEAL, D.Sc., F.I.C., and Associate (Industrial Chemistry). Pp. xv+219. London: Baillière, Tindall and Cox, 1920. Price 12s. 6d. net.

The publication of a series of books giving a general survey of the chemistry of industries will commend itself to all chemists, and to none more than to those

engaged in analytical chemistry. This is an age of specialisation, in which no chemical practitioner can possess a knowledge of all branches of chemical technology, and among the problems submitted to him, are frequently those connected with specific industries, so that unless he has some acquaintance with the requirements of these industries his reports may be practically without value to his clients. Dr. Rideal has set himself a heavy task to edit such a series of books, the success of which must depend entirely on securing the best specialists connected with each of the chemical industries.

The principal subjects with which the present volume is concerned are Starch and its Derived Sugars, Manufacture of Sugar from Sugar Cane and Sugar Beet, Sugar Refining, Malting, Brewing, Wine Making, Potable and Industrial Alcohol, Vinegar, Acetic Acid, Acetone, and Glycerin.

The sections contain some useful matters, but the text is marred by many errors and misstatements. This is the more regrettable in view of the fact that the book is written for the guidance of non-experts. Among these the following may be cited: Wheat is stated on one page to contain 55 to 65 per cent. of starch, and on another page its average starch content is said to be 68 per cent. It is not correct that the cheapest form of starch is that derived from potato, and the statement that "wheat starch" is used as a paste for "bill-posting," besides being inaccurate, may have the effect of giving undue prominence to the noble art cited. The title "Cane Sugar" and "Beet Sugar" for the sections dealing with the manufacture of sugar from cane and beet respectively is liable to revive the fallacy that sugar from the two sources differs. Goldthorpe barley is a broad-eared, not narrow-eared, two rowed barley; it belongs to the variety *Hordeum zeocriton*, not *Hordeum distichum*. The statement that by the malting process "the insoluble starch of the grain is converted into soluble fermentable sugar" is one we might expect to find in a book written in the eighteenth century.

Possibly the reason that the production of maltose in Belgium did not prove a commercial success was that the product was not entirely maltose, being made with malt extract. The statement that the percentage of maltose in commercial glucose (understanding thereby liquid glucose) generally exceeds that of dextrose is not the invariable rule.

Our criticism of this book must of necessity be severe, for it will undoubtedly fall into the hands of those who will believe implicitly everything they read in its pages. Whilst admitting, therefore, that some of the text has been carefully compiled, looking at the book as a whole we are forced to the conclusion that it does not fulfil its objects, and we hope the editor may in the near future, with the aid of experts, recast the book, omitting much detail which is not needed, and giving a succinct and accurate epitome of the chemical industries with which it is concerned.

A. R. LING.

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- Errata**: 1920:
- Page 210, line 11 from top, for "2549" read "2849".
 - Page 218, bottom line, for "69" read "169".
 - Page 262, line 24, delete "with."
 - Page 284, top line, for "saponifiable" substitute "unsaponifiable."
 - Page 339, line 4, for "1895, 19, 14" read "1894, 19, 141."
 - Page 450, line 9, add "ANALYST, 1920, 45, 139."
- Errata**: 1921:
- Page 22, lines for "Aluminium" read "Molybdenum."
 - Page 33, line 15, for "11" read "9."
 - Page 110, last line, for "1920" read "1921."
 - Page 111, line 10, for "1896" read "1895."
 - Page 131, line 21, delete "1909, 30."
 - Page 144, for "Onoperdon" substitute *Onopordon*.
 - Pages 143 and 374, for "acid-soluble" read "acid-insoluble."
 - Page 332, line 29, for "ANALYST" read "*J. Inst. Br.*"
 - Page 344, line 19, for "sulphite" read "sulphur trioxide."
 - Page 424, line 24, for "arsenious acid" read "arsenic."
 - Page 502, for "Dolt" read "Dott."

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THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

ORDINARY MEETING. DECEMBER 1, 1920.

HELD at the Chemical Society's Rooms, Burlington House, Mr. Alfred Smetham, President, in the chair.

A certificate was read for the first time in favour of Mr. W. R. Schoeller, Ph.D.

Certificates were read for the second time in favour of Messrs. Urban Aspey, Herbert Corner Reynard, B.Sc. (London), A.I.C., Edwin Burnhope Hughes, B.Sc. (London), A.I.C., Harry Jephcott, M.Sc. (London), A.I.C., Arnold Lees, A.I.C.

The following was elected a Member of the Society: Mr. T. K. Ghose, B.A., L.M.S. (Cal.).

The President referred to the regret with which the Council had received the resignation of Mr. Julian L. Baker from the Editorship of the ANALYST, a position which he had so ably filled for the last thirteen years, and congratulated him upon his appointment to the Editorship of the *Journal of the Institute of Brewing*.

The President announced that the Council had appointed Mr. C. A. Mitchell as his successor.

The following papers were read: "Estimation of Theobromine," by Raymond V. Wadsworth; "A New Process for the Estimation of Small Quantities of Chromium in Steels," by B. S. Evans, M.B.E., M.C., B.Sc., F.I.C.; "Some Notes on the Reactions between Fulminate of Mercury and Sodium Thiosulphate," by P. V. Dupré, M.B.E., A.C.G.I., F.I.C., and F. H. Dupré.



OBITUARY NOTICE.

J. W. GATEHOUSE.

MR. J. W. GATEHOUSE died on October 16, 1920, at Bath, at the age of seventy-nine, and we have to mourn the loss of one of our earliest members, whose death snaps one of the remaining links connecting the Society of Public Analysts in its early struggling days with the Society of to-day. He was also an original Fellow of the Institute of Chemistry.

Mr. Gatehouse was educated at Battersea College for the teaching profession, and subsequently became for a time science master at Hereford. Then, in 1868, he settled in Bath, where the rest of his long life was to be spent. For some years he carried on a private practice, in addition to teaching science at Downside College, near Bath, and other well-known schools. Long before the local Technical School was established he started classes for chemistry, botany, electricity, and kindred subjects, and many of his pupils were successful in the South Kensington examinations.

In 1877 Mr. Gatehouse was appointed Public Analyst for the City of Bath, an appointment which he held for over forty years, resigning in 1918. During the earlier years of his appointment he was engaged in many interesting chemico-legal cases, particularly in connection with the question of salt in beer, upon which he became an authority. A paper which he read upon this subject was published in the second volume of the ANALYST. In 1888 he was appointed Public Analyst for Wiltshire, and later became Electric Light Inspector under the Board of Trade and chemist under the Petroleum Acts.

Apart from his official duties, Mr. Gatehouse did much work upon the spectroscopic examination of the gases emanating from the Bath mineral waters, and was probably one of the first to discover that they contained helium, although his results were never published.

In recent years he specialised in the analysis and methods of testing calcium carbide and acetylene, having been official chemist to the Acetylene Association from its inception, and he devised a method and apparatus for estimating phosphorus and sulphur compounds in acetylene. This method was brought before the International Congress for Acetylene in 1894, and has been officially adopted as the standard method by many public bodies.



REACTIONS OF SUGARS AND POLYATOMIC ALCOHOLS IN BORIC ACID AND BORATE SOLUTIONS, WITH SOME ANALYTICAL APPLICATIONS.

By G. VAN B. GILMOUR, B.Sc. (Lond.), A.R.C.Sc.I., A.I.C.

THE property which boric acid solutions exhibit of becoming more acidic on addition of certain polyatomic alcohols and sugars was first pointed out by Klein in 1878 (*Bull. Soc. Chim.*, 29, 195). The explanation of such reactions was that combinations take place with the production of stronger acids. The subject has since been the source of a large amount of research, which has added very considerably to our knowledge of this type of reaction. It has been found that not only polyatomic alcohols and sugars form such combinations, but also that a large class of hydroxy compounds reacts similarly. The tendency of forming strong or weak complexes depends very largely on the position of the hydroxy groups, and Böseken has stated (*Rec. Trav. Chim.*, 1915, 34, 96-113) that when these groups are in the same plane and on the same side as the two carbon atoms to which they are bound, the position is favourable for condensation.

Early investigators stated that the polysaccharides failed to give positive reactions. Thomson (*J. Soc. Chem. Ind.*, 1898, 12, 432-433) obtained a positive reaction with cane-sugar, and the author has shown that lactose and maltose react in like manner. Thomson (*loc. cit.*) applied the reaction to a volumetric method of estimating boric acid. He found that in the presence of glycerol boric acid could be titrated to the compound NaBO_2 . He tried to replace glycerol by dextrose and cane-sugar, with unsuccessful results; in these cases the indicator showed up before sufficient alkali was added to convert the acid into metaborate. Vedam (*J. Pharm. Chim.*, 1898 [vi.], 8, 109-111) used mannitol in this titration; he considered that a sharper end-point was obtained with mannitol than with glycerol.

The increase in electrical conductivity imparted to boric acid solutions by the addition of polyhydroxy bodies is a measure of the stability of the compounds formed. The addition of mannitol has a very marked positive effect compared with glycerol, and this is reflected in the proportional parts of mannitol to glycerol required in the titration of a given quantity of boric acid; twelve times more of the latter than of the former must be present.

The author has been able to titrate boric acid successfully in the presence of lævulose, dextrose, and cane-sugar. Other very soluble sugars will probably act like these, but it was found impossible to carry out the titration with lactose. Weak combinations require a large excess of the hydroxy compound, otherwise the complex is hydrolysed before the proper end-point is reached. At 3 per cent. concentration dextrose, cane-sugar, lactose, and maltose have practically no influence on the acidity of boric acid compared with lævulose, which was found at this concentration to have a very powerful positive reaction, the effect on the acidity being of the same order as that of mannitol.

Agno and Valla (*Gazetta*, 1913, 43, 11, 163-174) from solubility measurements considered that boric acid and mannitol unite in equimolecular proportions. More

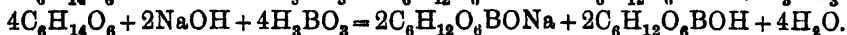
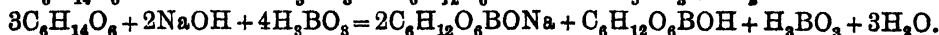
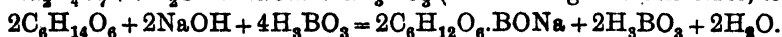
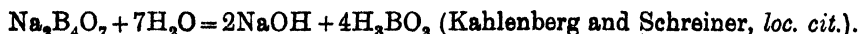
recently Grun and Nossioivitsch (*Monatsch.*, 1916, **37**, 409-423) have prepared salts of complex bodies formed by the union of boric acid with mannitol, sorbitol, and dulcitol. There can be no doubt that similar compounds are produced by combination with the sugars. The author has prepared the sodium derivative of a complex formed from l  vulose and boric acid by a method similar to that used by Grun and Nossioivitsch (*loc. cit.*). The constitution of this compound is being determined.

REACTIONS BETWEEN MANNITOL, BORIC ACID, AND CAUSTIC SODA.

An explanation of these reactions, which agrees with the observations of Kahlenberg and Schreiner (*Zeitsch. physikal Chem.*, 1896, **20**, 547-568) and of Magnanini (*Gaz. Chim.*, 428-440, **21**, 134-145), is as follows :

On addition of considerable excess of mannitol to a solution of boric acid, a union takes place in equimolecular quantities (Ageno and Valla, *loc. cit.*) to produce mannito-boric acid, $C_6H_{12}O_6 \cdot BOH$. When caustic soda is added to this, the sodium salt is formed, which is stable in acid solution, but in neutral or alkaline solution in the presence of mannitol it is readily hydrolysed into sodium metaborate and mannitol. The former is not stable in solution, and when liberated it immediately satisfies its residual valency by attracting whole mannitol molecules to form with them compounds like $NaBO_2 \cdot 3C_6H_{14}O_6$. In the volumetric estimation of boric acid the author has found that the minimum quantity of mannitol or l  vulose required to enable the acid to be titrated to the metaborate is approximately three molecules of either for one of the acid. These observations, together with the knowledge that the compound trimannitol sodium metaborate, $NaBO_2 \cdot 3C_6H_{14}O_6 \cdot 5H_2O$, had been prepared (Grun and Nossioivitsch, *loc. cit.*), led to the above explanation being put forward as to what happens when boric acid solutions are titrated in the presence of mannitol. Fructose seems to act exactly like mannitol, and, indeed, the type of reactions described might be considered general for polyhydroxy compounds that permit of the volumetric estimation of boric acid, the final state being one molecule of metaborate combined with one or more of the compound.

The acidity imparted to borax solutions by the addition of mannitol is due either to free boric acid, or to a mixture of boric acid and mannito-boric acid, or to mannito-boric acid alone, the final stage depending on the quantity of mannitol added. The following equations will illustrate this, in which the effect of adding two, three, and four molecular proportions of mannitol to one of borax is shown :



BORIC ACID IN MILK.

Farrington (*J. Amer. Chem. Soc.*, 1896, **18**, 847) found that when boric acid is added to fresh milk its acidity to phenolphthale  n is about four times as great as that of an aqueous solution containing the same quantity of the acid. Earlier in

this paper it has been pointed out that lactose combined with boric acid to produce a stronger acid, and if the table showing the effect of different quantities of lactose on the acidity of a solution of boric acid given in the experimental portion of the paper is consulted, it will be seen that the production of lacto-boric acid satisfactorily explains Farrington's observation.

Catalytic Action of Boric Acid.—Lowenthal and Lenssen (*J. prac. Chem.*, 1862, 85, 401) stated that boric acid retards the catalytic action of hydrochloric acid in the inversion of cane sugar. Their results were disputed by Arafura (*Mem. Coll. Sci. Eng., Kyoto*, 1909-10, 2, 229-236), who clearly showed the reverse to be the case. Arafura found that when the concentration of the hydrochloric acid is kept constant the accelerating effect of boric acid increases with its concentration, whereas the influence of boric acid on the catalytic action of hydrochloric acid is practically independent of the concentration of the latter acid.

It is easy to explain these observations of Arafura now that it is known that lævulose forms a strong lævuloso-boric acid. What actually happens is that the boric acid assists the inversion in two ways: firstly, it removes one of the products of inversion in the sense that its properties are altered; and, secondly, it forms a strong acid that assists the action of the hydrochloric acid.

Analytical Applications.—Lævulose can be used in the volumetric estimation of boric acid. On account of the high price of the pure sugar, its use would be prohibitive in such estimations. Invert sugar, on the other hand, is cheaply prepared, and is an excellent substitute. This is strongly recommended as a reagent to replace glycerol or mannitol. To titrate a given amount of boric acid, the minimum quantity of invert sugar required is about double the minimum of fructose.

The observation that cane-sugar and dextrose have practically no effect when present with lævulose, even in considerable excess, has led the author to attempt a method of estimating lævulose by finding to what point a known volume of standard boric acid can be titrated on the addition of a weighed quantity of the lævulose mixture, the acid's equivalent of lævulose being already determined. In the experimental portion of the paper a series of results is given; these are very encouraging, and it is thought that if the method is carefully standardised fairly accurate estimations can be made. A method such as this would be useful in the analysis of cane-syrup and honey, where the reducing sugar is practically all invert. The lævulose estimated in this way after the reducing sugar has been determined should give a figure approximately half that of the latter.

A method for estimating lævulose based on the measurement of the increase in electrical conductivity of boric acid solutions after the addition of lævulose might possibly give more accurate results than those obtained by the above titration method.

Suggested Use of Lævuloso-Boric Acid.—In medicine there are a number of well-known preparations in which the antiseptic properties of borax or boric acid are increased by means of glycerol. There is one preparation, "*Mel Boracis*," made from borax, glycerol, and honey, where the action of the honey has not been understood. The active principle in this preparation is lævuloso-boric acid, $C_6H_{10}O_6BOH$. This acid is very much more powerful than the corresponding glyceryl compound, and it is here suggested that invert sugar-syrup might be used more successfully as

6 GILMOUR : REACTIONS OF SUGARS AND POLYATOMIC ALCOHOLS

a basis for this type of preparation than glycerol, and, further, for the sake of economy, it should replace honey in "*Mel Boracis*."

Experimental.—Tables I. to X. show the effect of different amounts of polyatomic alcohols and sugars on the end-points in the titration of deci-molecular solutions of boric acid with deci-normal soda, using phenolphthalein as indicator, the end-point in each case being the first distinct pink coloration. When the alcohol or sugar was not neutral the acidity was determined and allowed for.

TABLE I.—GLYCEROL.

C.c. $\frac{1}{10}$ Molecule H_3BO_3 .	Grms. of Glycerol.	C.c. $\frac{N}{10}$ NaOH.
10.0	Nil	0.6
"	2.0	7.8
"	3.0	9.0
"	4.0	9.4
"	5.0	9.6
"	6.0	9.7
"	7.0	9.8
"	8.0	10.0

TABLE II.—MANNITOL.

C.c. $\frac{1}{10}$ Molecule H_3BO_3 .	C.c. of Water.	Grm. of Mannitol.	C.c. $\frac{N}{10}$ NaOH.
10.0	Nil	Nil	0.6
"	"	0.05	2.0
"	"	0.10	3.6
"	"	0.15	4.9
"	"	0.20	6.0
"	"	0.25	7.1
"	"	0.30	7.9
"	"	0.35	8.6
"	"	0.40	9.0
"	"	0.45	9.2
"	"	0.50	9.5
"	"	0.55	9.8
"	"	0.60	9.9
"	"	0.65	10.0
5.0	"	0.80	4.9
"	10.0	"	4.7
"	30.0	"	4.2

TABLE III.—LÆVULOSE.

C.c. $\frac{1}{10}$ Molecule H_3BO_3 .	Grm. of Lævulose.	C.c. $\frac{N}{10}$ NaOH.
10.0	Nil	0.6
"	0.05	2.0
"	0.10	3.6
"	0.15	4.6
"	0.20	6.0
"	0.25	6.7
"	0.30	7.6
"	0.35	8.1
"	0.40	8.6
"	0.45	9.0
"	0.50	9.3
"	0.55	9.5
"	0.60	9.8
"	0.70	10.0

The sugar used above contained approximately 90 per cent. of lævulose, so that the figures in the centre column are 10 per cent. too high.

TABLE IV.—DEXTROSE.

C.c. $\frac{1}{10}$ Molecule H_3BO_3 .	C.c. of Water.	Grms. of Dextrose.	C.c. $\frac{N}{10}$ NaOH.
5.0	10.0	Nil	0.3
"	"	0.3	0.9
"	"	1.0	2.3
"	"	3.0	4.0
"	"	5.0	4.6
"	"	6.0	4.9
"	"	7.2	5.0

TABLE V.—CANE-SUGAR.

C.c. $\frac{1}{10}$ Molecule H_3BO_3 .	C.c. of Water.	Grms. of Canesugar.	C.c. $\frac{N}{10}$ NaOH.
5.0	10.0	Nil	0.3
"	"	0.3	0.3
"	"	1.0	0.6
"	"	10.0	3.4
"	"	15.0	4.0
"	"	25.0	4.6
"	"	35.0	5.0
"	15.0	43.0	5.0

TABLE VI.—LACTOSE.

C.c. $\frac{1}{10}$ Molecule H_3BO_3 .	C.c. of Lactose.	C.c. $\frac{N}{10}$ NaOH.
5.0	Nil	0.3
"	5.0	1.7
"	10.0	2.2
"	15.0	2.6
"	25.0	3.0
"	50.0	3.5
"	80.0	3.8
"	140.0	4.1
"	280.0	4.4
"	450.0	4.0

Solution used contained 18 grms. of sugar in 100 c.c. of water.

TABLE VII.—MALTOSE.

C.c. $\frac{1}{10}$ Molecule H_3BO_3 .	C.c. of Water.	Grms. of Maltose.	C.c. $\frac{N}{10}$ NaOH.
5.0	Nil	Nil	0.3
"	10.0	1.0	1.6
"	50.0	10.0	3.1

More titrations with maltose were not carried out because, as the concentration increased, the solution became so dark in colour that the end-point could not easily be determined.

TABLE VIII.—MANNITOL AND DEXTROSE.

C.c. $\frac{1}{10}$ Molecule H_3BO_3 .	Grm. of Mannitol.	Grm. of Dextrose.	C.c. $\frac{N}{10}$ NaOH.
10.0	0.1	0.1	4.0
"	0.2	0.2	6.4
"	0.3	0.3	8.2
"	0.5	0.5	9.6

TABLE IX.—LÆVULOSE AND DEXTROSE.

C.c. $\frac{1}{10}$ Molecule H_3BO_3 .	Grm. of Lævulose.	Grm. of Dextrose.	C.c. $\frac{N}{10}$ NaOH.
10.0	0.1	0.1	4.0
"	0.2	0.2	6.5
"	0.3	0.3	8.1
"	0.5	0.5	9.6

The lævulose weights are 10 per cent. too high for the same reason as explained in Table III.

TABLE X.—MANNITOL, DEXTROSE, AND CANE-SUGAR.

C.c. $\frac{1}{10}$ Molecule H_3BO_3 .	Grm. of Mannitol.	Grm. of Dextrose.	Grm. of Cane-Sugar.	C.c. $\frac{N}{10}$ NaOH.
10.0	0.1	0.1	0.1	4.0
"	0.2	0.2	0.2	6.5
"	0.3	0.3	0.3	8.2
"	0.5	0.5	0.5	9.7

PREPARATION OF INVERT SUGAR REAGENT.

A satisfactory and rapid laboratory method is as follows :

Twenty-five grms. of cane-sugar are heated with 10 c.c. of water in a conical flask until completely dissolved ; the solution is boiled for a few minutes. Add 1 c.c. of $\frac{N}{2}$ HCl to the hot solution, and shake well without reheating. Now dilute, cool, and add 1 c.c. of $\frac{N}{2}$ NaOH, making the volume up to 50 c.c. This reagent is neutral and practically colourless, and 3 c.c. will enable 10 c.c. of $\frac{1}{10}$ molecule boric acid to be titrated. The minimum quantities of glycerol, mannitol, and invert sugar required to titrate 1 gram. of boric acid are respectively 128.0, 11.2, and 22.4 grms.

ESTIMATION OF LÆVULOSE.

It was first thought that lævulose might be estimated by determining the volume of a solution of the sugar that must be added to a known volume of an equi-molecular solution of caustic soda and boric acid containing phenolphthalein, so as to just discharge the colour. This proved unsatisfactory ; the colour faded fairly regularly until near the end-point, when the last trace of pink persisted to remain even after considerable additions of the sugar solution. The quantity of lævulose required to discharge the colour by this method is greater than that necessary to enable the same amount of boric acid to be titrated directly. The method finally arrived at was based on the results of the titrations shown in Tables III. and IX. It is carried out as follows :

10 REACTIONS OF SUGARS AND POLYATOMIC ALCOHOLS IN BORIC ACID

To a flask containing a weighed amount of the lævulose mixture are added 10 c.c. of a $\frac{1}{10}$ molecular solution of H_3BO_3 and 0.5 c.c. of 1 per cent. phenolphthalein. This is titrated to the first distinct pink by the addition of $\frac{N}{10}$ NaOH; on standing a little while the pink fades, but the first end-point is taken. The lævulose equivalent of the soda added is read off from a table of equivalents already calculated.

Table XI. below gives lævulose equivalents when mixtures such as syrup or honey are being analysed. In this table allowances have been made for the effect of other sugars present on the end-point. For volumes of $\frac{N}{10}$ NaOH intermediate to any two adjacent figures in the table it can be assumed that the lævulose equivalents increase proportionately.

TABLE XI.

Grm. of Lævulose	equivalent to	c.c. $\frac{N}{10}$ NaOH.
0.22		7.2
0.27		8.1
0.31		8.5
0.36		8.9
0.40		9.2
0.45		9.5

It is considered that best results are obtained when the $\frac{N}{10}$ NaOH readings lie between 6.5 and 9.5. A preliminary titration will indicate what quantities of the lævulose mixture are necessary to give readings between these limits.

Examples :

A Sample of cane-syrup containing 46.0 per cent. invert sugar.

B „ honey „ 75.9 „ „ „

Mixture.	Grms. of Mixture.	C.c. $\frac{1}{10}$ Molecule H_3BO_3 .	C.c. $\frac{N}{10}$ NaOH for Blank.	C.c. $\frac{N}{10}$ NaOH corrected for Blank.	Per Cent. Lævulose.
A	1.2	10.0	0.4	8.2	23.3
„	2.0	„	0.6	9.5	22.5
B	0.6	„	0.1	7.3	38.4
„	0.9	„	0.2	8.9	40.0

The percentage error in these estimations is of the order of 10 per cent., and is mainly due to the difficulty in determining the end-point.

MAYPOLE LABORATORY,
SOUTHALL.



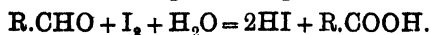
ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Reaction of Benzoic Acid and its Application to the Detection of Atropine, Cocaine, and Stovaine. M. Guerbet. (*J. Pharm. Chim.*, 1920, 22, 321-323.)—The reaction described depends on the conversion of the benzoic acid into a mixture of nitrobenzoic acids, reduction of these to aminobenzoic acids which are then diazotised, and the resulting diazonium compounds condensed with β -naphthol. The mixture of *o*-, *m*-, and *p*-naphthol azobenzoic acids formed has an orange-red colour. The test may be used for the detection of atropine, cocaine, and stovaine, or other substances containing a benzoyl group in their molecule. The details of the test are as follows: About 10 mgrms. of benzoic acid are mixed on a watch-glass with 5 drops of fuming nitric acid, and evaporated on a water-bath; the residue is treated with 1 drop of 10 per cent. stannous chloride solution, heated for three minutes, cooled, and 2 drops of 1 per cent. sodium nitrite solution are added. After a few minutes, 4 drops of a 1 per cent. β -naphthol solution in 10 per cent. ammonia are added, when a red orange-coloured precipitate is formed. If this final mixture is evaporated to dryness, the residue dissolved in concentrated sulphuric acid giving a red-violet solution which changes to yellow when the solution is poured into water.

W. P. S.

Estimation of Small Quantities of Dextrose. Perrier. (*J. Pharm. Chim.*, 1920, 22, 337-344.)—An iodimetric method is proposed for the purpose. Twenty c.c. of the sugar solution, containing a few mgrms. of reducing sugar, are treated with 1 c.c. of a 1.5 per cent. solution of crystallised sodium carbonate and 20 c.c. of $\frac{N}{100}$ iodine solution; after two hours the mixture is acidified with hydrochloric acid, and the excess of iodine is titrated with $\frac{N}{100}$ thiosulphate solution. From the quantity of iodine solution used 0.1 c.c. is deducted, and the remainder calculated into reducing sugar; the reaction proceeds according to the equation,



W. P. S.

Action of Hydrogen Peroxide on Flours. Marion. (*Comptes rend.*, 1920, 171, 804-806.)—The purity and quality of flour may be estimated by determining the action of its catalase upon hydrogen peroxide, the liberated oxygen being measured in a Niven ureometer. Four c.c. of 8 per cent. hydrogen peroxide, the acidity and oxygen content of which have been accurately estimated, are placed in the side tube of the nitrometer, into the main tube of which is introduced a thin paste of 2 grms. of the flour and 13 c.c. of water, rendered neutral with dilute sodium borate solution, which will add a volume of oxygen equal to 1.8 c.c. to the total liquid. The apparatus is immersed in water at 15° C., and inclined to bring the hydrogen peroxide into contact with the flour. The oxygen liberated in five minutes is collected

in a measuring tube attached to the apparatus, and is measured, after levelling, in the usual way. The following results were obtained with three grades of flour: No. 1, 1.75 c.c.; No. 2, 10.85 c.c.; No. 3, over 20 c.c. per gram. The amounts of cellulose separated from the same flours were 0.22, 0.76, and 6.40 per cent. respectively. Mixtures of 75 per cent. of No. 1 and 25 per cent. of No. 2 gave 5.95 c.c., and 25 per cent. of No. 1 with 75 per cent. of No. 2 gave 10.25 c.c. per gram. C. A. M.

Estimation of Water in Fats and Oils. H. Oertel. (*Chem. Zeit.*, 1920, 44, 854.)—On mixing in a porcelain dish 10 c.c. of the oil with a known quantity of a substance which evolves heat when moistened, such as a mixture of two parts anhydrous magnesium sulphate with one part kieselguhr, the temperature rise is observed by means of a thermometer graduated in tenths of a degree. The water content is read off from a table. Results by an independent observer agreed with the theory from 0 to 2.5 per cent. water, and showed 3.1 instead of 3 per cent.

O. E. M.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Estimation of Calcium in Blood-Serum. B. Kramer and J. Howland. (*J. Biol. Chem.*, 1920, 43, 35.)—One or 2 c.c. of plasma or serum is transferred to a platinum dish, evaporated on a water-bath, dried at 110° C., and gently ignited until completely incinerated. The ash is dissolved in $\frac{N}{10}$ hydrochloric acid and again evaporated and ignited, the residue being dissolved in 1 c.c. of $\frac{N}{10}$ sulphuric acid and transferred to a graduated test-tube. One drop of $\frac{N}{100}$ potassium permanganate solution is run in, when the pink colour should persist for at least one minute, after which 1 drop of 0.01 per cent. phenolsulphonephthalein and 1 drop of concentrated ammonia solution are added. The tube is heated on a water-bath to remove excess of ammonia, and while hot exactly 0.3 c.c. of $\frac{N}{10}$ oxalic acid in $\frac{N}{10}$ sulphuric acid is run in. If the solution is not acid in reaction at this point, $\frac{N}{10}$ sulphuric acid is added until the colour is lemon yellow. After heating for a few minutes the tube is cooled, 0.1 c.c. of saturated sodium acetate solution added, the contents well shaken, diluted to 2 c.c., and left to stand several hours. The mixture is filtered, and to 1 c.c. of the filtrate an equal volume of 20 per cent. sulphuric acid is added; the solution is then heated and titrated in daylight with $\frac{N}{100}$ potassium permanganate until a pink colour persists for thirty seconds. The results obtained are subject to a maximum error of 3 per cent.

T. J. W.

Estimation of Chlorides in Blood. V. C. Myers and J. J. Short. (*J. Biol. Chem.*, 1920, 44, 47.)—Three c.c. of blood or plasma are added to 27 c.c. of distilled water in a centrifuge tube, about 0.5 gram. of dry picric acid is added, the mixture stirred until of a bright yellow colour, then centrifuged at a moderate speed for a few minutes, and the clear supernatant liquid decanted. Twenty c.c. are pipetted into a second centrifuge tube, and 20 c.c. of 0.2904 per cent. silver nitrate solution, followed by 10 c.c. of acid ferric alum indicator, added. The precipitate is thrown down by centrifuging, and 20 c.c. portions of the supernatant liquid are removed and

titrated with standard ammonium thiocyanate, 2 c.c. of which is equivalent to 1 c.c. of the silver nitrate. By slightly modifying these conditions the same clear liquid from the picric acid precipitation may be used for the estimation of sugar, creatinine, and chloride. When using pure solutions, and also with known amounts of sodium chloride added to blood, the error due to the above method is less than 1 per cent., and the results agree closely with those given by the Austin-Van Slyke method (ANALYST, 1920, 45, 226).

T. J. W.

Estimation of Iodine in Blood and Animal Tissues. E. C. Kendall and F. S. Richardson. (*J. Biol. Chem.*, 1920, 43, 149 and 161.)—One hundred c.c. of blood or 100 grms. of solid tissue are placed in a nickel crucible, 10 c.c. of 30 per cent. sodium hydroxide solution added, and the crucible placed upon a hot plate until all water has evaporated. The mass is evenly heated over a Meker burner, and the gas supply adjusted so that all fumes cease to be evolved in less than twelve minutes, the cover being placed on as soon as frothing begins to subside. After cooling about 25 c.c. of water are added, the charred residue scraped from the side of the crucible, ground with a pestle, and boiled with the addition of a further 25 c.c. of water. After filtration the residue is again extracted with hot water containing a little sulphuric acid, and the filtrates mixed, after which 18 grms. of barium hydroxide are added, the solution filtered, sufficient sulphuric acid added to precipitate all barium, and filtration repeated through a pad of kaolin. The filtrate is boiled down to 15 or 20 c.c., transferred to a nickel crucible, and evaporated to dryness. The residue is fused, and from 5 to 10 mgrms. of potassium nitrate added, this being repeated until bubbles of gas cease to be produced on further addition. The cold melt is dissolved in 150 c.c. of hot water, and 5 c.c. of 20 per cent. sodium bisulphite solution and 2 drops of saturated aqueous methyl orange are added. When cold, 85 per cent. phosphoric acid is run in with shaking until the solution just becomes pink, when 5 c.c. of 20 per cent. phosphoric acid are added, and the solution boiled down to a volume of about 200 c.c. On cooling, bromine is added until the solution is yellow, and the solution again boiled for a period of five minutes after it becomes colourless, a few crystals of salicylic acid are dropped in, the solution cooled, and 5 c.c. of 20 per cent. phosphoric acid and 1 gm. of potassium iodide added, when the iodine liberated is titrated with $\frac{N}{250}$ sodium thiosulphate solution, using starch as indicator. Frequent controls should be made on all the solutions used. The method gives good results with solutions containing known amounts of iodine, but with blood the results show a loss of approximately 2·6 per cent. of the total iodine present.

T. J. W.

Colorimetric Methods for Estimation of Phosphorus in Urine and Blood. R. D. Bell and E. A. Doisy. (*J. Biol. Chem.*, 1920, 44, 55.)—*Inorganic Phosphorus*.—One to 5 c.c. of urine are measured into a 100 c.c. graduated flask, and 25 c.c. of water added. To a similar flask are added 5 c.c. of standard monopotassium phosphate solution (1 c.c. of which contains 0·1 mgrm. of phosphorus) and 25 c.c. of water. Five c.c. of molybdic acid solution containing 50 grms. of ammonium molybdate in 1,000 c.c. of $\frac{N}{1}$ sulphuric acid are added to each flask, followed

by 5 c.c. of a solution containing 20 grms. of hydroquinone and 1 c.c. of sulphuric acid per litre. After standing five minutes, 25 c.c. of a solution containing 16 per cent. of sodium carbonate and 3 per cent. of sodium sulphite are run in, the mixtures diluted to 100 c.c., well mixed, and left to stand from five to ten minutes. The colours of the solutions are compared in a colorimeter, and the amount of phosphate deduced.

Total Phosphorus.—One c.c. of urine, 6 or 8 drops of sulphuric acid, and 1 c.c. of nitric acid are placed in a hard glass tube, and cautiously heated until colourless and until nitrous fumes are no longer evolved. The residue is transferred to a 100 c.c. graduated flask with about 25 c.c. of water, and the estimation proceeded with as described above.

Organic Phosphorus.—Twenty c.c. of urine are run into a graduated 25 c.c. flask, made just alkaline with powdered barium hydroxide, diluted to 25 c.c., and filtered. Twenty c.c. of this filtrate are made faintly acid with dilute sulphuric acid, diluted to 25 c.c., filtered, and 10 c.c. of the filtrate (equivalent to 6.4 c.c. of urine) evaporated with sulphuric and nitric acids, the subsequent procedure being as described under total phosphorus.

The results obtained by these methods are slightly lower than those yielded by the uranium method, but agree well with the usual gravimetric results. For the estimation of phosphorus in blood similar methods are employed, after precipitating with trichloroacetic acid, as follows: Five c.c. of blood are mixed with 40 c.c. of distilled water, 5 c.c. of 20 per cent. trichloroacetic acid are run in during shaking, the whole diluted to 50 c.c., and filtered after standing ten minutes, 10 c.c. of the filtrate being used for the estimation.

T. J. W.

Estimation of Reducing Sugars in Blood, etc. O. Guillaumin. (*J. Pharm. Chim.*, 1920, 22, 378-390.)—The method of Folin and Wu (*ANALYST*, 1920, 227), with slight modifications, gives good results in the estimation of sugars in blood, except in the case of certain pathological processes which induce an accumulation of non-reducing sugars, which must be eliminated before applying the test. In the colorimetric comparison the intensity of the coloration increases more rapidly in the scale than the proportion of sugar, and the error thus caused is not negligible. This drawback of the method should be eliminated by making the comparison only under definite conditions corresponding with those under which the actual amounts of sugar have been estimated and recorded in the table.

C. A. M.

Toxicological Detection of Poisons containing Bromine. A. Damiens. (*Comptes rend.*, 1920, 171, 1020-1023.)—Bromine is a natural constituent of the lungs, 100 grms. of the latter containing 0.3 mgrm., and the ratio of bromine to chlorine is 0.00140. Examination of the lungs of men killed by poison gas consisting of bromine compounds showed that the bromine content of the lungs was higher than the above-mentioned figure; the largest amount found was 1.9 mgrms. per 100 grms. of lung substances, the bromine-chlorine ratio being 0.00386.

W. P. S.

Estimation of Catalase, Peroxydase, and Etherase in a Drop of Blood.

A. Bach and S. Zoubkoff. (*Comptes rend.*, 1920, 171, 967-969.)—A drop of blood is transferred by means of a 20 c.mm. capillary pipette into a flask containing 20 c.c. of water, and 1 c.c. of the mixture is used for each test. *Catalase*: 7 c.c. of water, 1 c.c. of the diluted blood, and 2 c.c. of neutral 1 per cent. hydrogen peroxide are shaken in a 75 c.c. flask, which is then heated for thirty minutes at 37° C. A control test is made at the same time with 1 c.c. of the dilute blood previously heated to boiling point. After the addition of 3 c.c. of $\frac{N}{1}$ sulphuric acid to each flask, the undecomposed hydrogen peroxide is titrated with $\frac{N}{10}$ potassium permanganate solution. The difference between the two results affords a measure of the catalase in the blood. *Peroxydase*: 7 c.c. of water, 1 c.c. of a 1 per cent. solution of pure guaiacol, 1 c.c. of the diluted blood, and 1 c.c. of 1 per cent. hydrogen peroxide are mixed in a test-tube, and the brownish-red coloration, which reaches its maximum after fifteen minutes, is estimated colorimetrically. As a colour standard a liquid is used, the coloration of which matches that of oxidised guaiacol. This is prepared by heating 5 grms. of egg albumin with 2 grms. of cobaltous chloride and 10 grms. of sodium hydroxide in 250 c.c. of water, and filtering the liquid. This is standardised against solutions containing 0.0005 to 0.001 grm. of guaiacol oxidised by means of an excess of vegetable peroxydase and hydrogen peroxide. The peroxydase is conveniently prepared by macerating 1 kilo of pulped radish with 500 c.c. of water, straining and filtering the extract, precipitating the peroxydase by means of alcohol, and redissolving the precipitate in 500 c.c. of water. With the addition of toluene the solution keeps indefinitely. *Etherase*: A solution of 0.2 grm. of potassium sulphoguaiacolate in 7 c.c. of water is mixed with 1 c.c. of 1 per cent. hydrogen peroxide, 1 c.c. of dilute peroxydase solution (1 : 20), and 1 c.c. of diluted blood (1 : 1,000), and the tube allowed to stand for thirty minutes at the ordinary temperature. The guaiacol liberated by the etherase is oxidised by the peroxydase and hydrogen peroxide, and is estimated colorimetrically in the same way as in the case of peroxydase. A control test is made, as before, with 1 c.c. of the dilute solution of blood previously heated to boiling-point. The examination of forty samples of blood by these methods gave the following results: *Catalase*: hydrogen peroxide decomposed by 1 c.mm. of blood, 17.17 to 18.78 mgrms. *Peroxydase*: 0.096 to 0.153 mgrm. of guaiacol oxidised. *Etherase*: 0.108 to 0.147 mgrm. of guaiacol liberated and oxidised.

C. A. M.

Formation of Gum Levan by Mould Spores. N. and L. Kopeloff and C. J.

Welcome. (*J. Biol. Chem.*, 1920, 43, 171.)—The spores of *Aspergillus sydowi* are thoroughly washed with sterile water, transferred to a sterile flask with 10 per cent. of chloroform, and heated at 62.5° C. for one hour. The suspension is thoroughly ground with sand, added to 500 c.c. portions of 10 per cent. sugar solution, and incubated at 40° C. for ten days. The clear solution is siphoned off, made slightly alkaline with sodium hydroxide, and the gum precipitated by the addition of five volumes of 95 per cent. ethyl alcohol. The precipitated gum is washed with alcohol, dissolved in water, and reprecipitated several times by alcohol, when it is dried on filter-paper, and finally on unglazed porcelain at 20° C. for eighteen hours. The

product is soluble to an opalescent solution in water, contains 0.16 per cent. of ash, gives a specific rotation of -40° in 0.25 per cent. solution, and on hydrolysis with concentrated hydrochloric acid the change in rotation produced indicates quantitative hydrolysis into levulose. Since the presence of levan in sugar solution renders the Clerget method of estimation inaccurate, the percentage of sugar present must be determined by the use of invertase, and Hudson's method (ANALYST, 1914, 39, 443) for the preparation of a solution of this enzyme possessing high activity is described. By a combination of the invertase method and the Clerget hydrolysis the amount of gum levan present in a sugar solution may be determined. A table is provided showing the reactions given by a number of reagents when added to an aqueous solution of levan.

T. J. W.

Reaction of Milk in Relation to the Presence of Blood-Cells and of Specific Bacterial Infections of the Udder. J. C. Baker and R. S. Breed. (*J. Biol. Chem.*, 1920, 43, 221.)—The authors examined the sensitiveness of brom-cresol purple, bromthymol blue, phenol red, rosolic acid, and Hoyberg's reagent to the acidity of milk in comparison with the P_H value determined electrometrically, and found the first-named indicator to give a more satisfactory series of changes of tint, especially with normal milk, than was shown by the other indicators. One hundred and twenty-four samples of milk from cows in three herds were examined, and the reaction, leucocyte and epithelial cell content, and the presence, both before and after incubation, of streptococci determined. From the results of this work the deduction is drawn that the number of leucocytes, epithelial cells, and streptococci present is roughly inversely proportional to the acidity of the milk, the primary cause of the abnormalities being in the majority of cases an infection with streptococci. The physiology of the results is discussed, and it appears probable that the decrease in acidity is due to the entrance of sodium bicarbonate from the blood.

T. J. W.

Estimation of Urea in Sera and Secretions. W. Mestrezat and M. P. Janet. (*J. Pharm. Chim.*, 1920, 22, 369-377.)—To obtain accurate results in the estimation of urea by the xanthidrol method (ANALYST, 1914, 39, 268, 362) it is necessary that the serum or other fluid should contain between 0.5 and 1.0 grm. of urea, preferably 0.5 grm., and that twice the amount of xanthidrol solution should be used. Ten c.c. of the serum diluted according to the amount of urea presumed to be present are mixed with 10 c.c. of Tanret's reagent (3.32 grms. of potassium iodide and 1.35 grms. of mercuric chloride in 20 c.c. of acetic acid and 60 c.c. of water) and centrifuged. The clear liquid (15 c.c.) is treated with an equal volume of glacial acetic acid, and then with 3 c.c. of a freshly-prepared 10 per cent. solution of xanthidrol in pure methyl alcohol added in three portions at intervals of ten minutes; the condensation of the xanthyl urea will be complete in three hours, and the crystals are then separated, washed with absolute methyl alcohol, dried in the water-oven, and weighed.

C. A. M.

Gravimetric Estimation of Albumin in Urine. G. Pegurier. (*Ann. Chim. anal.*, 1920, 2, 332-335.)—The urine is neutralised with acetic acid, filtered, and 50 c.c. of the filtrate are heated to boiling and allowed to cool for a few minutes; 5 c.c. of a solution of 10 grms. of phenol and 10 grms. of citric acid in 20 grms. of 95 per cent. alcohol are then added, the precipitated albumin is collected on a weighed filter, washed first with boiling water, then with a mixture of alcohol and ether, dried at 100° C., and weighed.

W. P. S.

Estimation of Total Nitrogen in Urine by the Dumas and Kjeldahl Methods. W. Mestrezat and M. P. Janet. (*Comptes rend.*, 1920, 171, 1019-1021.)—Comparative estimations of total nitrogen in urine showed that the Kjeldahl method always gave lower results than those found by the Dumas method, the deficiency varying from 0.09 to 0.42 grm. of nitrogen per litre of urine.

W. P. S.

Quantitative Method for the Determination of Vitamine in Connection with Determinations of Vitamine in Glandular and Other Tissues. F. K. Swoboda. (*J. Biol. Chem.*, 1920, 44, 531-551.)—The work of Williams (*ANALYST*, 1920, 45, 307) demonstrates that a substance of unknown structure, a constituent of yeast, is necessary in addition to the ordinary nutrients for the nutrition of yeast cells. This substance, Williams concludes, based upon identical occurrence and various properties, is identical with the beri-beri preventing vitamine. He further found that the growth of single cells of yeast may be used as a simple biological test for this vitamine. The work here presented shows that this vitamine, or, as McCollum calls it, the water-soluble B growth-promoting substance, can be quantitatively determined by a simple biological method, and from the experiments cited it is clear that the yeast cells, when grown on a synthetic medium containing sugar, asparagine, ammonium nitrogen, potassium, calcium, magnesium, sulphate, and phosphate, are dependent for their growth on a substance soluble in 95 per cent. alcohol and water, but of unknown chemical structure. This substance stimulates the growth of the yeast cell, or the multiplication of the cell, so that a unit weight of this substance is essential for the growth of a definite number of yeast cells (*cf. La Cellule*, 1901, 18, 313). This fact is made the basis of the present method for the quantitative determination of this substance, which in all probability is identical with the beri-beri curative vitamine. The biological test of Williams for the detection of small quantities of the water-soluble B vitamine was developed, and found to be of quantitative value. The method was applied, and the vitamine content of various organs determined. A summary of all determinations, giving the different vitamine numbers, is given. From the results of these determinations it is evident that this specific vitamine is present in large quantities in most of the organs of internal secretion which are of developmental importance, whereas it usually is present in very much lower concentrations in other organs examined. The liver and kidneys, however, are high in vitamine content. The tissues high in nuclear material, such as thymus and lymph gland, were found low in vitamine content. The fresh pancreas was found low in activity. It is suggested that high

content reported by others may be due to hydrolysis. The thyroid was the only organ in which an increase in concentration of the vitamine fraction caused a toxic action. Without proper dilution the vitamine activity would not be observed. These vitamine determinations afford additional evidence that the substance which increases the multiplication of the yeast cell is identical with the beri-beri curative vitamine.

H. F. E. H.

ORGANIC ANALYSIS.

Titrimetric Determination of Minute Amounts of Acetone. R. S. Hubbard. (*J. Biol. Chem.*, 1920, **43**, 43.)—The method adopted by the author is a modification of the one originally used by Messinger. Stock solutions of iodine in potassium iodide and of sodium thiosulphate are prepared, 1 c.c. of each being equivalent to 1 mgrm. of acetone. Water free from ammonia and other volatile matters is used for diluting these solutions to $\frac{1}{10}$, $\frac{1}{50}$, or $\frac{1}{100}$ of their original strength, and additional potassium iodide is added to the dilute iodine solutions to make the concentration of this salt approximately 3 per cent. These dilute solutions do not remain constant in strength for more than two days. To 50 or 100 c.c. of the acetone solution a known amount of iodine solution is added, the quantity and strength used depending upon the amount of acetone expected to exist in the solution, but in any case excess of iodine must be used. To the mixed solutions 2 c.c. of sodium hydroxide solution (200 grms. sodium hydroxide in 300 c.c. of water) are added, the mixture shaken, and allowed to stand ten minutes or longer, when 1 or 2 c.c. of 60 per cent. sulphuric acid is added, and the solution titrated with $\frac{N}{1000}$ or other known strength of sodium thiosulphate, using starch solution as indicator. Since the solutions to be estimated may contain substances, such as alcohol, liable to vitiate the results obtained by the above method, the author has worked out methods depending upon oxidation by sodium peroxide, sulphuric acid, and potassium permanganate or potassium dichromate, which destroy these substances, while the acetone present is unaffected. The results obtained by the above method are in good agreement with the actual amount of acetone present in aqueous solution.

T. J. W.

Estimation of Acetone in Expired Air. R. S. Hubbard. (*J. Biol. Chem.*, 1920, **43**, 57.)—The subject, fitted with a mask provided with an inlet valve for inhaled air, expired for ten minutes through two bottles arranged in series, each containing 75 c.c. of fresh 2.5 per cent. sodium bisulphite solution. Ten c.c. of 10 per cent. sodium hydroxide were added to each bottle, and the contents of each separately were washed into a Kjeldahl flask. The solution was distilled for ten minutes into a second Kjeldahl flask containing water to give a final volume of about 150 c.c. To the distillate 5 c.c. of 60 per cent. sulphuric acid and 0.2 grm. of potassium permanganate were added, and the solution again distilled to give about 100 c.c. of distillate. To this distillate 0.5 grm. of sodium peroxide were added, and the mixture distilled into a little water to give a volume of from 50 to 100 c.c. The acetone present in this final distillate was determined by the method given in the

preceding abstract or by the turbidimetric method, using the Scott-Wilson reagent (mercuric cyanide 10 grms., sodium hydroxide 180 grms., water 1,200 c.c.; the solution is shaken, and 400 c.c. of 0.7268 per cent. silver nitrate solution slowly run in, after which the solution should be kept at least one day before use). Quantitative determinations made by the above method show excellent agreement with the known amounts of acetone used. Normal adults exhale from 0.14 to 0.91 mgrm. of acetone per hour, but in cases of diabetes and exophthalmic goitre the amount increases to 5.24 and 15.0 mgrms. respectively.

T. J. W.

Analysis of Aromatic Nitro Compounds by Means of Titanium Trichloride. F. L. English. (*J. Ind. and Eng. Chem.*, 1920, 12, 994-997.)—Mononitro hydrocarbons are very resistant towards reduction by titanium trichloride, but the presence of positive or negative substituents, with the exception of chlorine, in the nucleus facilitates the reduction of the nitro group. Further, it would appear from the results of experiments carried out by the author that the position of the substituents with respect to the nitro group has no appreciable effect; for instance, *m*- and *p*-nitroaniline are reduced with equal ease, as are *o*- and *p*-nitrophenols, *o*- and *m*-nitro-*p*-toluidines, and two of the nitrosalicylic acid esters, whilst *o*- and *p*-nitrochlorobenzenes are about equally refractory. The essential point in the reduction is the excess of titanium trichloride used; the final concentration, after the mixture has been boiled, should be not less than 25 c.c. of $\frac{N}{20}$ titanium trichloride solution per 100 c.c. of total solution.

W. P. S.

Viscosity of Cellulose Ester Solutions. M. Deschiens. (*Chem. Trade J. and Chem. Engineer*, 1920, 67, 472.)—The determination of the viscosity of cellulose nitrate and acetate is of great importance, and the usual processes for measuring this property are unsuitable, because the evaporation of the solvent at the orifice of the viscosimeter leaves a film which blocks up the opening. The viscosimeter recommended is that of Ostwald, and is of a U-shaped pattern with the usual two bulbs, the base of the U, however, having right-angle bends. The standard for comparison adopted by the Allied Aviation Services is the viscosity of glycerol of 30° Baumé, at 15° C., the value of which is fixed at 100. The instrument is calibrated by means of glycerol under these conditions while almost entirely immersed in a thermostat at 15° C. If cellulose acetate is the ester under examination, the solution should be composed of 6 grms. of this material (preferably dried at 100° to 105° C.) and 100 grms. of acetone, great care being taken to see that solution is complete before use; if necessary, it may be centrifuged or filtered. Working with a solution of this strength, it was found that the time of flow was thirty seconds as against 190 for glycerol, and as this 190 is taken as being the 100 standard, the viscosity of the sample (x) = $\frac{190}{100} \times \frac{30}{x}$, where $x = 15.79$. Cellulose acetates of different origins, when dissolved in acetone, show figures varying from 42 to 10.5, and if intended for manufacture of aeroplane dopes, should lie between 10 and 30. Samples showing higher viscosity are preferable for the manufacture of plastic masses or celluloid.

H. F. E. H.

Analysis of Fabrics composed of a Mixture of Cotton and Wool. Duyk. (*Ann. Chim. anal.*, 1920, 2, 324-330.)—The following standard method has been proposed: From 10 to 15 grms. of the sample are dried at 100° C. and reweighed. The dry material is then treated with suitable reagents for the removal of dressing, filling, etc., washed, dried, and again weighed. The material, after this preliminary treatment, is heated at 90° C. for twenty minutes with 2 per cent. sodium hydroxide solution, then washed first with water, next with acidified water, again with water, dried at 100° C., and weighed. The loss in weight corresponds with the amount of wool present, and the proportions of cotton and wool are calculated on the original substance. If silk is also present in the fabric, the latter is treated with zinc chloride solution (sp. gr. 1.65) before the cotton and wool are separated as described; the zinc chloride dissolves the silk, but leaves the cotton and wool unaffected.

W. P. S.

Chia Oil. H. A. Gardner. (*Chem. Trade J.*, 1920, 67, 674-675.)—The seeds of the Mexican plant, chia, yield a yellow oil resembling linseed oil in odour and taste. A sample of the seeds contained 33.83 per cent. of oil. The filtered oil had the following values: Sp. gr. at 15.5° C., 0.9338; $[n]_D^{25^\circ \text{C.}} = 1.4855$; acid value, 0.6; saponification value, 192.2; iodine value, 196.3; and unsaponifiable matter, 0.8 per cent. In the raw state the oil dried somewhat slowly, and showed pronounced "crawling"—i.e., tendency to coalesce in drops at points, leaving the previously coated surface bare. This was prevented by heating the oil for fifteen minutes at 500° F., and in this condition the oil was superior to linseed oil as a drying oil. The oil-cake contained: Oil, 10.5; nitrogen, 3.6; and phosphoric anhydride, 1.5 per cent.

C. A. M.

Jelly Strength of Gelatins and Glues. S. E. Sheppard, S. S. Sweet, and J. W. Scott, jun. (*J. Ind. and Eng. Chem.*, 1920, 12, 1007-1011.)—A torsion dynamometer is described for determining the jelly strength of gelatin and glue under pure shear of moulded cylindrical test pieces. Both the breaking load and percentage twist at break are measured; the product of breaking load $\times$ twist, divided by the cross section of the test piece, is taken as the jelly strength. There is no simple relation between concentration of gelatin and the jelly strength; hence, the jelly strength values determined for a single arbitrary concentration give a very arbitrary comparison of the jelly strengths. There appears, therefore, to be no definite relationship between the jelly strength at a given concentration and the glue-joint or tensile strength of a dry glue joint.

W. P. S.

Determination of Sulphur Monochloride in Mustard Gas Mixtures. W. A. Felsing, S. B. Arenson, and F. J. Kopp. (*J. Ind. and Eng. Chem.*, 1920, 12, 1054.)—Twenty-five c.c. of approximately $\frac{N}{10}$ sodium iodide solution and 10 c.c. of carbon tetrachloride are run into a stoppered flask, which is then accurately weighed. By means of a pipette 2 c.c. of the mustard gas-sulphur chloride mixture is added rapidly, and the flask again weighed. A known excess of standard sodium thiosulphate solution is run in, and the excess titrated with standard iodine solution,

using starch as the indicator. The number of c.c. of sodium thiosulphate used multiplied by its equivalent of sulphur monochloride ($S_2Cl_2 = I_2$) gives the content of the latter substance in the sample. The accuracy of the method is within 0.5 per cent. in concentrations of 20 to 30 per cent. of sulphur monochloride, and within 0.1 per cent. for small proportions. The method is reliable for concentrations as low as 0.04 per cent. of sulphur monochloride. T. J. W.

INORGANIC ANALYSIS.

Analysis of Light Aluminium Casting Alloys. R. M. Berry. (*J. Ind. and Eng. Chem.*, 1920, 12, 998-1000.)—One grm. of the alloy is treated with 20 c.c. of 25 per cent. sodium hydroxide solution, and the mixture diluted to 300 c.c. as soon as effervescence ceases; this is necessary to prevent tin passing into solution. The insoluble portion, containing the tin, copper, lead, iron, nickel, part of the manganese, and traces of aluminium, is collected on a filter, and washed ten times with 1 per cent. sodium hydroxide solution. The filtrate contains 99 per cent. of the aluminium and zinc and the remainder of the manganese; it is neutralised with formic acid, 25 c.c. of the acid are added in excess, the mixture heated to boiling, and treated with hydrogen sulphide. The zinc sulphide is collected, washed with hot water, dissolved in hydrochloric acid, the solution boiled to expel hydrogen sulphide, and the zinc then titrated with potassium ferrocyanide solution. *Tin*.—The portion of the sample insoluble in sodium hydroxide solution is heated with 10 c.c. of nitric acid, the mixture then diluted with 30 c.c. of hot water, the metastannic acid collected, washed with hot water, dissolved in ammonium sulphide solution (to separate silica, traces of iron, lead, etc.), the solution filtered, and the tin sulphide reprecipitated from the filtrate, collected, ignited to SnO_2 , and weighed. *Copper and Lead*.—The filtrate from the metastannic acid is diluted to 150 c.c., and the copper and lead are deposited electrolytically, using a current of 1 ampere and 2.5 volts; about forty-five minutes are required for the deposition of the lead. Two c.c. of dilute sulphuric acid (1 : 1) are then added, and the electrolysis continued for a short time. The electrodes are at once removed from the solution, washed twice with water and once with alcohol, and dried, the cathode at 80° C. for fifteen minutes and the anode at 210° C. for thirty minutes. The increase in weight of the cathode gives the copper, whilst the increase in weight of the anode is the PbO_2 . *Iron*.—The electrolyte from the copper and lead estimation is treated with 3 grms. of ammonium chloride, rendered ammoniacal, boiled, and the ferric hydroxide (with a trace of aluminium hydroxide) is collected, redissolved in dilute hydrochloric acid, and the iron titrated with permanganate solution after reduction with stannous chloride. *Nickel*.—The filtrate from the iron is rendered just acid with hydrochloric acid, boiled, dimethylglyoxime is added, the mixture then rendered very slightly ammoniacal, and, after one hour, the precipitate is collected, washed with hot water, then with alcohol, dried at 110° C., and weighed. The weight multiplied by 0.2031 gives the amount of nickel present. *Magnesium*.—This is estimated by the phosphate method in the filtrate from the glyoxime precipitate. *Manganese*.—One grm. of the alloy is treated with 25 per cent. sodium hydroxide solution, an excess of nitric acid

is then added, together with a few crystals of silver nitrate, the mixture is boiled, oxidised with persulphate, cooled, and the permanganate formed titrated with arsenite solution. *Silicon*.—One grm. of the alloy is dissolved in a mixture of nitric and sulphuric acids, a few drops of hydrochloric acid are added, the solution is evaporated to dryness, the residue taken up with hydrochloric acid and water, and the silica collected, ignited, and weighed. The ignited silica should be treated in the usual way with hydrofluoric acid.

W. P. S.

Analysis of Aluminium, Cobalt, and Chromium Alloys. J. R. Camp and J. W. Marden. (*J. Ind. and Eng. Chem.*, 1920, 12, 998.)—A suitable quantity of the alloy is heated with a small quantity of *aqua regia*, and the solution then evaporated with the addition of 3 c.c. of sulphuric acid, and heated until sulphuric acid fumes appear. After cooling, the solution is diluted to 100 c.c., transferred to a pressure bottle, saturated with hydrogen sulphide, the bottle then closed, heated in a boiling water-bath for one hour, allowed to cool, and the molybdenum sulphide collected and washed. The filtrate is again saturated with hydrogen sulphide to make certain that all the molybdenum has been precipitated. It is advisable to redissolve the precipitate and repeat the precipitation. The molybdenum sulphide is finally ignited at a dull red-heat in a muffle furnace for two hours and weighed as MoO_3 . The filtrate from the molybdenum sulphide is boiled to expel hydrogen sulphide, sodium peroxide is added, the mixture boiled, and the precipitated cobalt hydroxide collected, dissolved in dilute hydrochloric acid, again precipitated with peroxide, collected, ignited, and weighed as Co_3O_4 . The filtrate from the cobalt is acidified with sulphuric acid, boiled to decompose peroxide, and the chromate in solution then titrated with ferrous ammonium sulphate solution. A sample of the alloy analysed by the authors yielded molybdenum, 41.52; cobalt, 50.89; and chromium, 7.56 per cent. The alloy known under the name of "Stellite" has a similar composition; it is extremely hard, and is used for making cutting tools.

W. P. S.

Estimation of Traces of Bromine in Organic Substances. A. Damiens. (*Comptes rend.*, 1920, 171, 799-802.)—The organic material is dried at 100° to 105° C., with the previous addition of a small amount of potassium hydroxide in toxicological work, the dry substance pulverised with 5 parts of potassium nitrate and 10 parts of sodium carbonate, and the mixture heated in a silver crucible in a muffle furnace, but without allowing the mass to fuse. It is then treated with water, the solution filtered after twenty-four hours, and the filtrate divided into two portions. Iodine is estimated in one of these by treating the acidified liquid with an excess of silver nitrate, and leaving it for twenty-four hours in the dark. The precipitate is then washed, suspended in 10 c.c. of water, and a current of chlorine introduced for fifteen minutes in the cold, after which 1 c.c. of sulphuric acid is added, and the current of chlorine continued for five minutes at 100° C. Finally, the chlorine is expelled by a current of air, the liquid centrifuged, and the separated solution treated with a few drops of sulphur dioxide solution, 2 c.c. of chloroform, and an excess of 5 per cent. sodium nitrite solution. If iodine be found, it is estimated colorimetrically

in the chloroform or (if more than 0.5 mgrm. is present) by a volumetric method, after complete extraction of the liquid with chloroform. The second portion of the original aqueous solution is neutralised, acidified with nitric acid (1 c.c. excess), treated with excess of $\frac{N}{10}$ silver nitrate solution, boiled for ten minutes, and placed for three hours on the water-bath. The precipitate is separated the following day, washed, and reduced by mixing it with 3 to 4 c.c. of water, 3 drops of sulphuric acid, and a fragment of zinc. If less than 1 mgrm. of iodine was found the solution is filtered, reduced, the silver washed, and the washings added to the solution in which the bromine is estimated by the method of Denigès and Chelle (*ANALYST*, 1913, 38, 119). If more than 1 mgrm. of iodine be present, the filtrate and washings are neutralised with ammonia, diluted to 40 c.c., and distilled with 1 gm. of ferrous ammonium sulphate, so as to leave a residue of 10 c.c., in which the bromine is then estimated. By this modification of the method 0.005 mgrm. of bromine can be estimated.

C. A. M.

Catalytic Decomposition of Alkaline Sodium Hypobromite Solution by Copper Sulphate. P. Fleury. (*Comptes rend.*, 1920, 171, 957-960.)—In view of the fact that salts of nickel, cobalt, and copper decompose hypochlorites and hypobromites with the liberation of oxygen, and that copper is sometimes present as an impurity in sodium hydroxide, the ordinary method of estimating urea by means of alkaline sodium hypobromite solution may give erroneous results. This catalytic action of copper may be entirely inhibited by the action of iodine, whether present in the form of iodide or iodate. Hence it is advisable, when a solution of hypobromite is required for the estimation of small quantities of urea, to add about 0.1 per cent. of potassium iodide to solution (a) of the following reagent: (a) Sodium hydroxide solution, 55 c.c. diluted to 100 c.c. with water. (b) Bromine, 8.5 c.c.; potassium bromide, 50 grms.; water, 80 to 82 c.c. Equal parts of the two solutions are mixed just before use.

C. A. M.

Estimation of Copper in Sugar Analysis by Means of Potassium Thiocyanate and Potassium Iodide. G. Bruhns. (*Zeitsch. anal. Chem.*, 1920, 59, 337-359.)—The following procedure is recommended for the iodimetric estimation of copper, since it effects a considerable saving in the quantity of potassium iodide required: Twenty c.c. of Fehling's solution and 20 c.c. of the sugar solution are mixed, heated to boiling, boiled for exactly two minutes, and cooled rapidly. Five c.c. of potassium thiocyanate iodide solution (containing 0.65 gm. of potassium thiocyanate and 0.1 gm. of potassium iodide) are added, followed by 10 c.c. of 6.5 N sulphuric acid, and the mixture is at once titrated with thiosulphate solution (34.4 grms. per litre). A control titration is made at the same time, using the same quantities of reagents, but omitting the heating, and the difference between the quantities of thiosulphate solution required for the two titrations is a measure of the sugar present. Tables are given showing the quantities of invert sugar corresponding with quantities of thiosulphate solution varying from 0.8 to 19.6 c.c. in differences of 0.1 c.c.

W. P. S.

Volumetric Estimation of Hydrosulphite. R. Formhals. (*Chem. Zeit.*, 1920, **44**, 869.)—The hydrosulphite, 0.5 grm., is dissolved in about 50 c.c. of boiled water, and titrated with $\frac{N}{10}$ potassium ferricyanide solution until the indicator, a few drops of 10 per cent. ferrous ammonium sulphate solution, gives a blue coloration. The sulphurous acid is without effect in the cold. The standard solution is best adjusted against pure hydrosulphite. O. E. M.

Microchemical Reactions of Iodic Acid. A. Bolland. (*Comptes rend.*, 1920, **171**, 955-957.)—*Thallium nitrate* at first produces crystalline needles which appear colourless to the naked eye, but black under the microscope. In the second phase the crystals unite in groups of crosses with straight arms. They have an average size of 40 μ . The reaction is capable of detecting 1 part of iodic acid in 5,000. *Silver nitrate* gives an amorphous product, which crystallises from ammonia solution in rhombic plates (up to 100 μ), united in groups of four or six. Sensitiveness of test, 1 : 5,000. *Barium chloride* gives a precipitate of straight or curved needles (up to 300 μ) grouped in twos or fours. Sensitiveness, 1 : 2,500. *Strontium acetate* forms needles and fine prisms (100 μ) grouped together, frequently in the form of a cross. Sensitiveness, 1 : 300. *Calcium acetate* forms colourless monoclinic octahedra of mean size 150 μ . Sensitiveness of test, 1 : 300. *Rubidium chloride* produces rectangular plates, which have the appearance of pentagons or hexagons. The crystals (25 μ) show a strong refraction. Sensitiveness of test, 1 : 300. In the case of concentrated solutions of iodic acid, caesium sulphate, potassium chloride, sodium acetate, ammonia, and magnesium chloride, also form characteristic crystals, the sensitiveness of the tests ranging from 1 : 20 to 1 : 80. C. A. M.

Sensitive Colour Reaction of Phosphates and Arsenates. G. Denigès. (*Comptes rend.*, 1920, **171**, 802-804.)—On treating a very dilute solution of ammonium molybdate with a stannous chloride reagent, a blue coloration due to a basic salt of molybdenum is obtained. This reagent is prepared by heating 0.1 grm. of tin foil with 2 c.c. of hydrochloric acid and 1 drop of 3 per cent. copper sulphate until solution is complete, diluting the liquid to 10 c.c. with water, and decanting it from any deposit. Acidification of the dilute molybdate solution with hydrochloric or sulphuric acid checks, and at a certain limit inhibits, the reduction by stannous chloride, but if at this point a solution of a phosphate is added, a stable blue coloration, due to the formation of a complex molybdic phosphate, is produced. The test is capable of detecting 0.02 mgrm. of phosphorus in the form of phosphate, and can be applied in the presence of organic matter. A complex blue arsenate is formed under the same conditions, and the reaction may be used for the colorimetric estimation of arsenic deposits obtained in Marsh's test. Conversely the reaction may be used for the detection of tin or molybdenum. C. A. M.

Phosphomolybdic and Phosphotungstic Acids. H. Wu. (*J. Biol. Chem.*, 1920, **43**, 189.)—The author discusses the history, modes of formation, chemical properties, and composition of the various phosphomolybdic and phosphotungstic

acids, and suggests their application in analytical chemistry. The "mixed acid" solution used is prepared by dissolving 100 grms. of sodium tungstate and 25 grms. of sodium molybdate in 700 c.c. of water, adding 50 c.c. of 85 per cent. phosphoric acid, 100 c.c. of hydrochloric acid, and boiling under a reflux condenser for eight hours.

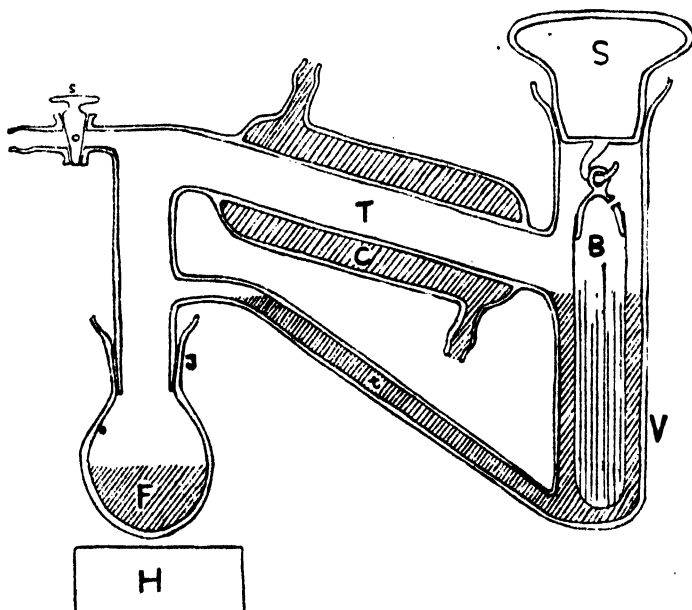
Detection of copper: To the solution under examination are added a few drops of 5 per cent. potassium cyanide solution, hydrochloric acid in sufficient amount to give an acid reaction, and a few drops of the above "mixed acid" solution. The solution is rendered alkaline by the addition of sodium carbonate, and a blue colour is produced if copper is present even in traces.

Detection of phosphoric acid: 1 or 2 c.c. of 2 per cent. ammonium molybdate, a few c.c. of 10 per cent. potassium iodide, 1 c.c. of 10 per cent. sodium bisulphite solution, and finally 1 to 2 c.c. of hydrochloric acid are added to the solution to be tested, which is then mixed and allowed to stand twenty minutes. On making alkaline by the addition of saturated sodium carbonate, a blue colour indicates the presence of phosphoric acid. This test is sensitive to 1 part of phosphorus in 1,000,000 parts of water, and may also be made the basis of a method for the colorimetric estimation of phosphoric acid. The "mixed acid" solution is also suggested as an indicator in oxidation or reduction titrations.

T. J. W.

APPARATUS, ETC.

Apparatus for Continuous Dialysis or Extraction. H. Mann. (*J. Biol. Chem.*, 1920, **44**, 207-209.)—The apparatus consists essentially of a flask, a condenser, and a vessel so arranged that the system may be evacuated, and that liquid



placed in the apparatus may be caused to circulate by means of continuous evaporation and condensation. Substances that will pass into solution are extracted and

concentrated under thermal and chemical conditions favourable for the preservation of labile and easily-decomposed compounds. Fig. 1 is a drawing of the apparatus. The flask, F, is equipped with a ground joint, J, provided with a mercury cup. H is a heating unit suitable for heating the flask, F. An incandescent bulb may be used. *s* is a stop-cock attached to a short tube. T is a condenser tube, and C is the condenser. V is a vessel fitted with a ground stopper, S, provided with a mercury cup. B is a bag of collodion, cloth, or other material suspended in the vessel, V. *t* is a narrow tube, through which fluid in V can flow back into F. The method of operation is as follows: The substance to be extracted or dialysed is placed in the bag, B, which is suspended in the vessel, V. Enough of the desired solvent is introduced into the vessel, V, so that it overflows through the tube, *t*, into the flask, F, partly filling the flask. The apparatus is then exhausted to the desired degree through the tube which is fitted with the stop-cock, *s*, and this stop-cock is then closed. The condenser tube, T, is now cooled by means of a stream of water circulating through the condenser, C, and the flask, F, is warmed by the heating unit, H. There results a continuous evaporation of the fluid in the flask, F, and a continuous condensation of this evaporated fluid in the condenser tube, T. The condensed liquid flows into the vessel, V, causing the level of the fluid in V to rise, so that fluid flows through the small tube, *t*, back into the flask, F. Thus the vessel, V, is continuously receiving a supply of freshly distilled solvent, and discharging its content into the flask, F, in which flask continuous evaporation of the fluid and concentration of non-volatile matter are taking place. The apparatus has the following advantages: A small amount of solvent can be used for the extraction of a large amount of material; this extraction can be made as complete as desired, and the substances extracted are at the same time concentrated. The apparatus, being operated at reduced pressure, is especially suitable for the extraction of labile substances, which are destroyed either by oxidation or by high temperature. With water as the solvent and the apparatus well exhausted, it is not necessary to warm the flask, F, more than 10° to 15° C. above room temperature. By using a collodion bag or other suitable dialysing membrane, it is possible to extract and concentrate the diffusible substances in plant and animal tissues—as, for example, the amino-acids of blood, muscarin, and other thermolabile alkaloids from fungi—and thus to obtain many substances of pharmacological importance.

H. F. E. H.



REVIEWS.

INDUSTRIAL GASES. By HAROLD CECIL GREENWOOD, O.B.E., D.Sc., F.I.C.
Pp. xvii + 371. London: Baillière, Tindall and Cox, 1902. Price 12s. 6d.

This book, forming one of a series on chemical industries, edited by Dr. S. Rideal, is a useful addition to chemical literature. Excepting a good translation of Claude's "Air liquide, Oxygène et Azote," and articles in dictionaries, very little was to be found on this subject in the English language.

One can only regret that the gifted author should have died at the early age of thirty-two, almost on the eve of the publication of his work; the foreword by Dr. J. A. Harker indicates plainly the loss to science and industry occasioned thereby.

The work is divided into an Introduction and three parts dealing respectively with (1) The Gases of the Atmosphere; (2) Hydrogen, Carbon Monoxide, Carbon Dioxide, Sulphur Dioxide, Nitrous Oxide, Asphyxiating Gases; (3) Gaseous Fuels.

The Introduction gives an excellent account of the physics of gases, as well as of the equilibrium of gas reactions.

Part I. deals chiefly with the separation of oxygen, nitrogen, and the rare gases of the atmosphere; there is, in addition, a section on ozone. This part of the book contains, as might be expected, descriptions of liquefying plant; the explanations are clear, and the illustrations well chosen.

More than half of Part II. is devoted to hydrogen, nearly fifty pages being assigned to the industrial preparation of this gas. This division is certainly justifiable in view of the great importance of pure hydrogen in synthesising ammonia, hardening fats, or, in fact, in any process in which hydrogen is brought into combination with the aid of a catalyst.

Part III., dealing with gaseous fuels, only takes up sixty-four pages. The reviewer would have liked to have seen a few woodcuts illustrating the construction of the plant employed, even if these illustrations had been quite diagrammatic in character. It must be admitted, however, that the author succeeded in utilising the sixty-four pages to great advantage, and the exposition of "Fundamental Principles" is excellent.

Both Subject and Authors' Indexes appear to be thorough, and the value of this useful book is enhanced by the lists of references given at the end of many of the sections.

J. T. HEWITT.

SOLUBILITIES OF INORGANIC AND ORGANIC SUBSTANCES. By ATHERTON SEIDELL, Ph.D. Second Edition. Pp. xxii + 843. Price 45s. net.

Even a casual survey of this book arouses in the reader two feelings: one of gratitude to the author for his achievement, the other of thankfulness that the compilation of such stupendous compendia of physical constants is no longer almost exclusively the work of other than English-speaking authors. The publication of books of this type, of the value and, indeed, of the indispensability of which there can be no question, should be the duty of the scientific societies, and one feels, there-

fore, all the more admiration for the individual whose patient, persevering labour has resulted in the production of this book. Twenty-three of the best-known scientific journals from the year 1900 onwards have been searched page by page for solubility data, and the tables of contents of twenty-five other journals have been consulted. The mass of information obtained proved too voluminous to form a supplement to the first edition, so the whole book has been revised and enlarged, the new matter including the sources of the solubility data (given in an Authors' Index), freezing-point data, the methods of determining solubilities, and the calculation of solubility data in any desired terms. The order of arrangement of the inorganic and the organic substances is entirely alphabetical, which renders the book very easy to consult for information of the solubility of any desired substance. With this arrangement a Subject Index appears at first sight superfluous. Its value, however, is twofold. A substance such as phenol, for example, is mentioned many times, sometimes as solute, sometimes as solvent; as solute it appears under "P," whilst references to its use as solvent are given in the Subject Index. Again, synonymous names appear, not in the alphabetical list, but only in the index, where, for example, under "acetphenetide" reference is given to "phenacetin." Great care has been taken in gathering the solubility data, and, wherever possible, the composition of the solid phase in equilibrium with the solution has been given. No attempt has been made to give information about the melting-point data of mixtures of metals (alloys) or minerals, the freezing-points of very dilute solutions, the solubility of gases in molten metals, the "solubility" of metals in solvents due to chemical action, solid solutions, or data for substances of unknown or variable composition.

The book will undoubtedly find a place in scientific institutions and in the laboratories of chemical factories where the value of research work is appreciated.

CLARENCE SMITH.

AN INTRODUCTION TO THE PHYSICS AND CHEMISTRY OF COLLOIDS. By EMIL HATSCHEK. Third Edition. Pp. 116. London: J. and A. Churchill, 1920. Price 4s. 6d. net.

This book is already so well and favourably known from the earlier editions that a brief notice of the appearance of a third edition is sufficient. Although more extended accounts on the subject are now available in English, Mr. Hatschek's work will hold its own on account of its practical character and the clearness and accuracy with which it is written. It can be heartily recommended to all who desire a brief and trustworthy introduction to this important and rapidly extending branch of science.

GEORGE SENTER.

A LABORATORY MANUAL OF ELEMENTARY COLLOID CHEMISTRY. By EMIL HATSCHEK. Pp. 135. London: J. and A. Churchill, 1920. Price 6s. 6d. net.

No one is better fitted than Mr. Hatschek to produce the first work on practical colloid chemistry. He has not only tested personally the methods described, but has experienced the difficulties of students in carrying them out, and as a result his book will prove indispensable to all interested in the study of this important subject.

The book is divided into nineteen chapters, and full directions are given for the preparation and examination of emulsoids, suspensoids, emulsions, and sols and gels of various types. The processes of dialysis, ultrafiltration, cataphoresis, and optical methods of examination of colloids are illustrated, as well as the electrolyte precipitation of suspensoid sols, the mutual precipitation of suspensoid sols, and the "protection" of colloids. Measurements of viscosity and the phenomenon of adsorption are also described. Most of the experiments can be carried out with the ordinary apparatus of a chemical laboratory, and the descriptions are clear and accurate. A considerable number of illustrations add to the value of the book. References to recent literature are given at the end of each section.

All advanced students of chemistry would be well advised to perform a selection of the experiments described in this book.

GEORGE SENTER.

THE MANUFACTURE OF SUGAR FROM THE CANE AND BEET. By T. H. P. HERIOT.
London: Longmans, Green and Co., 1920. Price 24s. net.

The manufacture of sugar is such a varied process that it is impossible in a limited space to deal with all the different methods and apparatus employed. The author sets out with the idea of giving an account of the scientific principles on which successful practice is dependent. Separate chapters are devoted to the study of the two chief sources of sugar—viz., the beetroot and sugar-cane—and to the necessary differences of treatment due to the differences in structure of the plants and in the composition of their juices. Refining of the raw sugars and disposal of the residues are also considered. It is pointed out how the beet-sugar producer has been greatly in advance of the cane-sugar producer in scientific investigation and the application of scientific methods. More work is now being done in research in some directions in the sugar-cane industry in Java and other parts of the world, but, generally speaking, the British cane-sugar producer has not realised the importance and absolute necessity of research in all departments connected with his work if he is to keep up with his competitors.

The book is largely a compilation from standard works on sugar manufacture, references to which are given in the text. In many cases, too much space is taken up in elaborate details which might, with advantage, have been better employed in more careful elucidation of the principles involved in the operations.

The language is often very loose and inexact. Split infinitives are frequent, and there is an irritating use of commas, and especially of semicolons, where they are not wanted.

A few examples of inaccuracies may be indicated: "The seed is the reproductive organ." "Each flower contains stamens and pistils, and are termed perfect flowers." Pollination and fertilisation are taken as synonymous. The whole of the botanical portion is weak, and requires revision.

In the preface the carbonatation process is properly so called, but throughout the text this is shortened to "carbonation." To be consistent, "sulphitation" might equally well have been called "sulphation."

It would be well for "decolourising" to be used for the process of removal of

colouring matters, and "bleaching" for their *oxidation or reduction* to colourless compounds, instead of making "bleaching" cover both processes.

On pp. 245-246 "heating-surface" is employed to mean three different things. First it is the *metal partition* between the steam and the liquid to be evaporated, then it is the *area of heated metal* in contact with the liquid, and finally it is the *metal surface* in contact with the steam.

On p. 277 the definition of the coefficient of supersaturation should be the inverse of what is stated.

On p. 285 the right-hand table does not add up to 100 as given.

It is surely a very loose statement (pp. 295-296) that *centrifugal force acts only on one of the constituents, solid or liquid, in a centrifugal machine.*

The illustration of the brasmoscope on p. 316 shows the vacuum and temperature in cm. and degrees C. respectively, while in the description inches and degrees F. are employed. Throughout the book there is no consistency in the units adopted, shillings per ton and dollars per 100 kilos being quoted, and degrees F. and degrees C., kilos and tons, are used indiscriminately.

On p. 324 the ratio of 100 dry solids : cane sugar is given as the purity, whereas it should be cane sugar $\times 100$: dry solids, as stated on p. 147. On p. 338, 1 part CaO to 1 part of sugar is said to form the soluble monocalcium saccharate, while on the next page 1 part of lime to 1 of sucrose is said to cause tricalcium saccharate to be precipitated.

The principles which underlie the methods of chemical control and analysis of sugar products are given, but the details are said to lie outside the scope of the work. Such an expression, however, as "88 per cent. net analysis" does not convey any definite idea to the non-expert without some information as to its meaning.

The book is too detailed in many parts for the general reader, and does not contain sufficient original matter or fresh treatment of the subject for the practical sugar manufacturer.

The printing is good, and for a book of reference it lies open conveniently flat, but the binding leaves much to be desired.

H. MAIN.

THE ANALYST.

OBITUARY NOTICE.

JOHN RUFFLE, F.I.C.

MANY of the older members of this Society will mourn the death, on November 19, at the age of nearly seventy-eight, of John Ruffle, who joined the Society in its early days as a very welcome adherent to its ranks, when, unlike now, those who did not actually hold official appointments were in a decided minority.

Ruffle, who was born in Suffolk, went to Germany as a lad, and spent some years on an estate in the Rhineland. Having thus acquired a taste for agricultural pursuits, he returned to England in 1861 and became a student in the Royal Agricultural College at Cirencester, where he took his diploma and, being attracted especially by the chemical side of his studies, he became and remained for some years an assistant in the laboratory of the consulting chemist to the Royal Agricultural Society, viz., the late Dr. Augustus Voelcker, his old chemical professor. From there he went as chemist to the fertiliser factory of Messrs. Jas. Gibbs and Co. at Silver-town, where he remained until 1884. He then practised as a consulting chemist on his own account for two or three years in the City but relinquished his practice to take up the post of chemist to the "Cape Copper Company and Butenferry Chemical Works" at Neath in South Wales, where he remained until 1896. In 1897 he became a chemist in the works of Messrs. Allen and Hanburys, Ltd., at Ware, and this post he held until his death, which occurred almost suddenly.

Although his interest in both pure and applied chemistry was always keen and led him into a good deal of investigational work, his publications were not many. A paper on the "Examination of Copper Sulphate," read before a meeting of the Society of Public Analysts in 1893, was published in the ANALYST of that year. His most notable contribution to analytical literature, however, was his paper on the "Estimation of Nitrogen by Combustion, including Nitro-Compounds," published in the *Transactions of the Chemical Society* for 1881, in which he demonstrated that the admixture of sulphur with the material to be analysed and of sodium thio-sulphate with the soda-lime used resulted in the complete conversion of nitric nitrogen into ammonia, the process being especially designed for use in the analysis of complex fertilisers containing nitrates as well as organic and ammoniacal nitrogen. Although the soda-lime combustion process, whether in its original form or as modified by Ruffle, has now given way to the more easy and rapid Kjeldahl moist combustion method, with its modifications due to Jödelbauer, Gunning, and Arnold, the process of Ruffle has not been surpassed in its accuracy. In the *Journal of the Society of Chemical Industry* for 1887 two notable papers were printed embodying

the results of general work on the composition of superphosphate and compound manures derived therefrom, in which he showed that the then conventional methods of expressing the results of analyses of such materials were based upon erroneous conceptions. The titles of these papers were "Correct Analysis of Plain and Ammoniated Superphosphates" and "Moisture in Superphosphate." Various other contributions from his pen, some based on his own work and some in criticism of the work of others, also appeared from time to time in the same journal and in the *Chemical News*, but these do not now call for especial mention.

Ruffle's disposition was sensitive and retiring and very gentle, and he was much loved by his early colleagues and contemporaries. Living, however, in geographical retirement for many years past, and but rarely visiting his old scientific haunts, he would be personally unknown to most of those of the younger generations of our members.

He was married in 1871, and became a widower in 1906. He had two children, a daughter who died in 1898, and a son, Mr. Hugh W. E. Ruffle, who survives him.

BERNARD DYER.



PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE ESTIMATION OF THEOBROMINE IN COCOA AND ITS PRODUCTS.

By RAYMOND V. WADSWORTH.

(*Read at the Meeting on December 1, 1920.*)

THE methods available at present for the estimation of theobromine are all, for various reasons, far from being satisfactory, yet they are so numerous that the introduction of another seems to necessitate an apology. As Whympster remarks (Allen, "Comm. Org. Anal.," vol. vi.), "The recorded values of theobromine are very variable and untrustworthy, arising from the lack of any very accurate and reliable method of estimation." It is this unreliability that has urged the author to search for some method which will be simple, efficient, and rapid, and at the same time give concordant results in the hands of different workers. At the present time, with most of the methods suggested, agreement between different workers is difficult to obtain. The process described below is given in the belief that it overcomes these difficulties.

PREVIOUS METHODS.—The methods suggested up to the present day may be roughly divided into four groups:

A. Those based on the precipitation of the alkaloid from the acid extract of cocoa by phospho-tungstic or phospho-molybdic acids, and the estimation of the

alkaloid in the precipitate, by the nitrogen content (Weigmann), by ignition (Wolfram and Legler), or by solution in a solvent (Kunze, Monthulé).

B. The cocoa, with or without a previous acid treatment, is extracted with water and an alkali; MgO in the method of Mulder, Bell, Savini, Trojanowski, Frommes, Beckurts, and Dekker; CaO in the method of Zipperer; Ba(HO)₂ in the method of Eminger; PbO in the method of Süss; and the alkaloid extracted by chloroform from the dried filtrate (Dekker, Frommes, Savini, Zipperer, Süss, Beckurts), or from the residue obtained by evaporation of the whole to dryness (Mulder, Bell, Eminger, Trojanowski). In the methods of Trojanowski and Bell the solvent used is alcohol.

C. The alkaloid is extracted directly with a solvent, such as chloroform (Diesing), or chloral alcoholate (Kreutz) from the dry material.

D. The alkaloid is extracted from the *wet* material by a solvent, phenol-chloroform (Maupy and Débourdeaux).

CRITICISM.—A criticism of most of these methods has been made by W. E. Kunze (ANALYST, 1894, 11, 194), but there are other criticisms which the author would like to offer—criticisms which the grouping of the methods facilitates.

A. The precipitation by phospho-tungstic or phospho-molybdic acids has been stated to be incomplete, and certainly in the hands of some operators it gives low results. The precipitate itself is one difficult to free from impurities owing to the nature of the materials precipitated together with the alkaloid, this operation taking a whole day to perform. The estimation of the theobromine by the nitrogen content of the precipitate obviously gives high results, as does the ignition of the aqueous or amylic-alcohol extract. The extraction of the alkaloid by chloroform has the drawbacks of low solubility, etc., mentioned below (under B.), though the actual alkaloid obtained is generally purer than that obtained by the methods of the second group.

B. The solution of the alkaloid in water, as before, but the precipitation of objectionable substances by alkalies, as magnesia, lime, baryta, or lead, has the disadvantage again of poor filtration, and also of incomplete precipitation. It has therefore been thought by some (Mulder, Bell, Eminger, Beckurts, Trojanowski) that filtration is unnecessary. In either case the result is much the same, other bodies besides the alkaloid being extracted by the solvents, as is apparent from the colour of the resulting alkaloid and the colour of its solution in water. The solubility of theobromine in chloroform, as has been previously pointed out by the author (ANALYST 1920, 45, 133), is very low; consequently a large quantity of solvent or long extraction is necessary, and so even slightly soluble bodies have every chance of being dissolved with the alkaloid. In addition to this, the extraction from a perfectly *dry* material is very slow, and in the author's opinion never complete. This seems to have been recognised by Savini and Frommes, who employed small quantities of ammonia in the solvent. Alcohol, as used by Bell and Trojanowski, which contains the small quantity of water found necessary for the extraction, is a better solvent for the alkaloid, but also has the great disadvantage of dissolving more foreign bodies than chloroform. Kunze obtained about four times more extract (theobromine?) by this than by any other method.

Many authors (Frommes, Kreutz, Eminger, etc.) have insisted on a treatment with acid being necessary as a preliminary to the estimation of theobromine,

suggesting that a compound is present from which the alkaloid cannot be obtained without this previous treatment. The compound is generally regarded as a glucoside. Kreutz (ANALYST, 1909, 34, 20) remarks that "it is impossible to separate the free theobromine from that portion of the alkaloid existing as a glucoside." Fritsch ("Fabr. du Chocolat," p. 264) suggests a combination of theobromine with tannin. If Fritsch's assumption be correct, and this compound be formed or present, an acid treatment would be of no help, as this compound is not decomposed by acids. It is, however, readily soluble in boiling water, and easily decomposed by magnesia, baryta, lime, or lead by the precipitation of the tannin. The author has never been able to obtain an increase in the yield of theobromine by this acid treatment; in fact, combining this with Dekker's method, usually from 0.1 to 0.25 per cent. lower yield was obtained, but the alkaloid was obviously purer, being much whiter in colour. Any compound present—and a definite proof of its existence is still wanting—is decomposed by water and the compounds mentioned above. Even unfermented beans do not give a higher result after the acid treatment. The only way a higher result can be obtained is by a breaking down of the cellular tissue of the material by the acid, in cases where very fine powders are not used, or where no water is used, as with the methods of Kreutz and Diesing.

It has been stated (Diesing and Süss) that lime, and to a less extent magnesia, decomposes some of the theobromine on prolonged heating, and it is accepted that in the case of Zipperer's low results this was due to decomposition of the alkaloid by the lime. With magnesia the author finds a slight decomposition, reducing the percentage on prolonged heating at 100° C. by 0.1–0.2 per cent., but with short heating the result is not affected. Thus in the second group it is fairly certain that the small reduction in the percentage is brought about by the long treatment with alkalis. In the author's hands the acid treatments have never shown any decomposition of the alkaloid.

C. A direct and complete extraction of the alkaloid with a solvent from the dry cocoa is impossible. The quantity of material other than alkaloid extracted is very considerable, amounting, in the case of fermented cocoas, to 50 per cent. of the total extract, whilst the actual theobromine extracted is seldom more than half that really present in the material. The alkaloid is only slowly given up. After 80 hours' extraction the author found between 0.06 and 0.10 per cent. again extracted in 24 hours, though after 120 hours no more could be extracted. Again, the low solubility of the alkaloid in chloroform is a drawback, and, as Maupy has shown, water is absolutely necessary to the complete extraction. To extract the remainder of the theobromine, Kreutz, after a direct extraction with chloroform or ohloral alcoholate and ether, gives an acid treatment, neutralises, dries, and extracts the alkaloid again with chloroform. This second extraction has the drawbacks mentioned above, and the method as a whole gives high results.

D. To overcome these difficulties, Maupy (ANALYST, 1897, 22, 191) suggested and worked out a method, using phenol-chloroform as the solvent, and mixing the solvent to be extracted with water. Without water this method gives much the same results as Diesing's. Phenol-chloroform is a good solvent, but inconvenient to work with, and, unfortunately, dissolves out other bodies which precipitate with the

theobromine, thus giving high results. The amount of other matter depends upon the cocoa used, and varies between 0.2 and 0.5 per cent. With the object of purifying the product, Débourdeaux (*J. Soc. Chem. Ind.*, 1917, 612) added to the process a long and tedious method of purification. Dekker objected to the method on the ground that the alkaloid was not completely precipitated from solution. This only occurs on those rare occasions when a *very large* amount of foreign matter has come through, and in those cases precipitation is complete after standing overnight.

It is obvious from the above review that none of the methods as yet suggested gives satisfactory results. Even the methods most commonly in use (Dekker and Kunze) have very serious drawbacks. Cocos of different origin have been found to give very variable amounts of foreign matter in solution with the alkaloid, particularly in the case of fermented and unfermented cocos of the same origin. This, perhaps, accounts for the higher results obtained by some workers with fermented cocos, a result which led them to suggest the formation of theobromine during the process of fermentation. The method described below gives a pure extract in all cases, and is rapid and easy to work.

The solvent used is tetrachlorethane (acetylene tetrachloride), which, as has been previously pointed out (*ANALYST*, 1920, 45, 133), is one of the few good organic solvents for theobromine. Used on the damp cocoa, it has just the same drawbacks as phenol-chloroform, but with a previous treatment with magnesia this difficulty is overcome, and the complete extraction of the pure alkaloid is obtained.

NEW METHOD.—If the cocoa bean is to be extracted, it is necessary to remove the fat with petroleum spirit (of b.-pt. below 80° C.). If, however, cocoa or cocoa shell is being used, this preliminary extraction is not necessary.

Ten grms. of the material are placed in a small porcelain dish and well mixed with 2–3 grms. of freshly calcined magnesia. This mixture is very carefully triturated with 14 c.c. of water until every particle is wetted. The dish containing the damp mixture is then placed on the water-bath for half an hour to dry it partially. The mass should be mixed at intervals to prevent any part becoming perfectly dry. At the end of the half-hour the mixture is well triturated and transferred to a flask of about 250 c.c. capacity; 150 c.c. of tetrachlorethane are added, and the whole is boiled under a reflux air condenser for half an hour. It is then filtered, *while almost boiling*, into a second flask. The residue is transferred, together with the filter paper, to the first flask, 120 c.c. of tetrachlorethane added, and the mixture boiled for twenty to thirty minutes. The liquid is then filtered into the second flask, and the residue washed twice more as before. The united washings are distilled through an air condenser until reduced to 3 or 5 c.c. The residue is cooled, and 60–70 c.c. of ether (methylated, of sp. gr. 0.720) are added, the whole well mixed, and allowed to stand overnight. The precipitate is then collected on a tared filter paper, and washed with ether, dried at 100° C., and weighed. To this weight is added 0.004 grm., as representing the amount of theobromine dissolved in the 70 c.c. of ether used in the precipitation and in the washings.

Details and Precautions.—The material should only be extracted with petroleum spirit in those cases where it is impossible, owing to the fat present, to wet the material with water—*i.e.*, cocoa beans or mass. When extracted, even with petroleum

spirit, there is always a very slight loss of the alkaloid. The author has proved during his work that theobromine is slightly soluble in cacao butter, which accounts for the loss, also found by Süss, during extraction. Süss suggested that the loss was due to its being carried over mechanically, for he found theobromine absolutely insoluble in cacao butter. The author has found that the cacao butter of commerce is already saturated with both caffeine and theobromine at about 80° C. Süss's failure therefore to detect the solubility was possibly due to his using commercial fat. The amount of water necessary for the proper wetting of the material varies between 9 and 20 c.c. according to the substance used. Cocoa powder generally requires 14 c.c., but for cocoa shell 20 c.c. are required. The water should be added a few c.c. at a time, and the mixture well triturated after each addition. When the mixing, which should be very thorough, is finished, the whole mass should be easily compressible into a firm cake. The most convenient mixer for this process is a glass rod, with one end flattened. During the half-hour's drying on the water-bath the material should soon become easily broken up, and granular in appearance. When this happens the stirring should become a little more frequent. At the end of the half-hour the whole should be very thoroughly mixed again, so as to assure that every particle is damp, as this is very necessary for the extraction. When transferring the material to the flask a little of the powder will adhere to the basin owing to its dampness; this can be wiped off with a piece of moist filter paper, and the paper dropped into the flask. The filtered solvent from the extraction should, when hot, be perfectly clear and almost colourless, and, when a high percentage of alkaloid is expected, a fourth washing should be given. The final distillation of the solvent should be taken as low as possible, the alkaloid commencing to precipitate out some minutes before the end of the distillation. In practice it is found best to distil the majority of the solvent from each filtration whilst the next boiling is taking place. This enables a smaller flask to be used, and facilitates the final distillation, which should be carried as far as possible. After precipitating the alkaloids with ether, it will be found in most cases that one hour's standing is sufficient to ensure a complete precipitation of the theobromine. However, if the supernatant liquid be even slightly turbid, it should be allowed to stand overnight, and, where time is no object, this is the best method. The resulting alkaloid should be white and clean. It sometimes contains a small amount of insoluble matter and ash, amounting together to about 0.004 grm. on the average. For greater accuracy, therefore, it is advisable, after weighing, to wash the filter with boiling water to dissolve the theobromine, and to weigh the residue, care being taken that the washing is very thorough, as the alkaloid is not very easily dissolved. The filtrate is evaporated to dryness, ignited, and the ash weighed. Both are deducted from the first weighing.

The table on p. 37 shows a few results of the amounts of insoluble matter and ash obtained in experiments on different materials.

The magnesia treatment is too short to allow of any decomposition of the theobromine; it decomposes any compound of the alkaloid which may be present, and it precipitates or converts into an insoluble form all the bodies which would otherwise dissolve in the solvent. The presence of the water allows of a rapid and complete

solution of the alkaloid by swelling up the material, and thus allowing the egress of the theobromine from the otherwise almost impermeable cells through the medium of the water. The method thus combines the advantages of groups B and D described above, while overcoming their drawbacks. The time taken for a complete estimation on a cocoa powder may be as short as five or six hours, and two estimations can be performed as easily as one in the same time. The process estimates only the theobromine, which is the principal alkaloid of cocoa; the caffeine is left in solution when the theobromine is precipitated with the ether.

	Weight of Precipitate.	Weight of Insoluble Substances.	Weight of Ash.
	Grm.	Grm.	Grm.
A.	0.226	0.002	0.002
B.	0.238	0.002	0.001
C.	0.225	0.003	0.002
D.	0.199	Nil.	0.001
E.	0.220	Nil.	0.001

PURITY OF THE THEOBROMINE OBTAINED.—With concentrated sulphuric acid at 100° C. the product gives only a very light-brown coloration. The ash and the amount insoluble in water are practically nil. The nitrogen content estimated by the "absolute" method is 31.07 per cent., which is equivalent to 99.9 per cent. theobromine, the theoretical result being 31.11 per cent. The nitrogen in theobromine cannot be estimated accurately by the Kjeldahl method owing to the alkaloid volatilising without decomposition. By this method the nitrogen in pure theobromine varies between 27 per cent. and 29.5 per cent. according to the speed of heating and other details of manipulation. Thus the estimation of nitrogen in cocoa by the Kjeldahl method never includes the whole of that present in the theobromine. The percentage of silver in the salt obtained from the alkaloid is found to be 37.4, which is equal to 99.5 per cent. theobromine, the theoretical yield being 37.59 per cent. By the precipitation of the theobromine as the silver salt caffeine has been shown to be absent.

Thus the alkaloid obtained by the above method has a purity of 99.5 to 99.9 per cent.

The work was carried out in Messrs. Cadbury Bros' Research Laboratory, and I wish to thank the firm for their permission to publish the results.



A NEW PROCESS FOR THE ESTIMATION OF SMALL QUANTITIES OF CHROMIUM IN STEELS.*

By B. S. EVANS, M.B.E., M.C., B.Sc., F.I.C.

THE following process was devised to meet a need that arose for the estimation of small percentages of chromium in steels. Existing processes cannot be relied upon for the estimation of amounts less than, say, 0.05 per cent., whilst the amounts requiring to be estimated were about 0.01 or 0.02 per cent.

The principle of the method is oxidation of the chromium to chromate in the presence of ammonium phosphate, pouring the boiling solution into excess of boiling sodium hydroxide solution, making up to a known volume, filtering, and determining the chromate colorimetrically.

The details of the process are as follows: 4 grms. of the sample are weighed out and brushed into a flask of capacity about 700 c.c.; 30 c.c. of 1:3 sulphuric acid and 20 c.c. of water are added, and the mixture heated till the steel is dissolved, more water being added if the ferrous sulphate begins to crystallise out; 5 to 10 c.c. of nitric acid are then run in to oxidise the ferrous sulphate and the carbon, and boiling continued till the red fumes are dispelled. About 25 grms. of ammonium phosphate are then added (the exact amount is immaterial, and the ordinary moist crystals are used) and 250 c.c. of water (and more nitric acid if ferric phosphate separates), and the whole heated to boiling again. When boiling, a saturated solution of potassium permanganate is added two or three drops at a time, till excess of permanganate is present, as shown by a permanent precipitate of manganese dioxide or by a permanent red colour, as the case may be, and then a further excess of about 12 drops, and the boiling is continued for fifteen minutes. While the oxidation of the chromium is progressing, 120 c.c. of 20 per cent. sodium hydroxide solution are placed in a large beaker, about 14 drops of saturated permanganate added, and the whole boiled for some minutes; if the purple of the permanganate be entirely changed to green during this time, more should be added, so that at the end there is still a purplish tinge. When the solution of the sample has boiled for fifteen minutes, the sodium hydroxide solution is taken off the plate, about 10 c.c. of 5 per cent. manganese sulphate solution added to destroy the permanganate, and the acid solution of the sample slowly poured into it, with constant shaking, the flask being gripped with rubber-covered tongs. The resulting liquid is then poured into a 500 c.c. measuring flask, a drop of it tested with litmus paper to ensure its being strongly alkaline, and it is cooled down; if, when tested, it is found to be acid, it should be made strongly alkaline with sodium hydroxide; this does not prejudice the result. When cool, 10 c.c. of strong acetic acid are added (the liquid should be again tested to make sure that it is acid) and it is made up to the mark, shaken well, and allowed to settle for a short while; it is then filtered through dry paper into a dry flask, the first few c.c. being rejected.

All colouring matter, other than chromate, has now been removed from solution, with the exception of a small amount of nickel or cobalt if those metals be present.

* Communication from the Research Department, Woolwich.

If chromium be absent the solution is practically water-white. In the absence of nickel and cobalt the estimation of the chromium is proceeded with as follows: 100 c.c. of the filtrate are placed in one Nessler glass and a little less than that volume of distilled water in another; 20 c.c. of 1:3 sulphuric acid are added to each and $\frac{N}{100}$ potassium dichromate run into the blank glass till the colours match, the volume before the final matching being made up to the same volume as that of the test solution.

If nickel or cobalt be present they interfere somewhat with the colorimetric estimation, and the procedure must be modified as follows: The 100 c.c. of filtrate are measured into a flask, instead of a Nessler tube, and heated to boiling; the flask is then removed from the plate, and sodium hydroxide solution is added until a precipitate separates, a large excess being avoided. The liquid is then cooled, filtered through a small paper filter into a Nessler glass, 20 c.c. of 1:3 sulphuric acid added, and the colorimetric estimation finished as above.

There is usually a very small blank: this may be determined, for each separate estimation, as follows: After the tubes have been matched, a drop (or quantity just sufficient) of dilute ferrous sulphate solution is added to each, and they are compared again. The liquid in the standard tube is now practically water-white, whilst that in the sample tube may be very faintly coloured, and, if so, the depth of the colour must be determined in terms of $\frac{N}{100}$ potassium dichromate solution by filling a fresh Nessler tube with 100 c.c. of water and 20 c.c. 1:3 sulphuric acid and running in $\frac{N}{100}$ potassium dichromate solution till the colours match. This blank is then subtracted from the original burette reading; it is generally about 0.25 c.c., and is of a barely perceptible colour. Of course, this only applies to quantities of chromium so small as not appreciably to colour the solution in the reduced form; with larger amounts it is sufficiently accurate to ignore the blank, or to assume that it is 0.25 c.c.

If x be the number of c.c. $\frac{N}{100}$ $K_2Cr_2O_7$ used in the titration, and y be the number of c.c. $\frac{N}{100}$ $K_2Cr_2O_7$ used in the blank, then $(x - y) \times 0.0217 =$ percentage of chromium in sample.

The following results were obtained with electrolytic iron to which different quantities of chromium had been added. In all experiments throughout this work the chromium added was measured as $\frac{N}{100}$ potassium dichromate solution, and reduced before being added to the iron solution:

No. of c.c. $\frac{N}{100}$ $K_2Cr_2O_7$ added to 4 Grms. Fe.	No. of c.c. $\frac{N}{100}$ $K_2Cr_2O_7$ recovered per 4 Grms. Fe.	No. of c.c. $\frac{N}{100}$ $K_2Cr_2O_7$ required for Titration of 100 c.c.	Blank. c.c.	Per Cent. of Chromium added.	Per Cent. of Chromium recovered.
1.00	$0.17 \times 5 = 0.85$	0.42	0.25	0.0043	0.0037
2.00	$0.35 \times 5 = 1.75$	0.60	0.25	0.0087	0.0077
3.00	$0.55 \times 5 = 2.75$	0.80	0.25	0.0130	0.0120
4.00	$0.80 \times 5 = 4.00$	1.05	0.25	0.0174	0.0168
5.00	$1.00 \times 5 = 5.00$	1.35	0.35	0.022	0.022
10.00	$2.00 \times 5 = 10.00$	2.35	0.35	0.044	0.044
20.00	$4.00 \times 5 = 20.00$	4.35	0.35	0.087	0.087
40.00	$7.60 \times 5 = 38.00$	7.95	0.35	0.174	0.167

Some tests were made in a similar manner with electrolytic iron to which varying quantities of some other metals, and small proportions of chromium, had been added:

Metal added.		No. of c.c. $\frac{N}{100}$ $K_2Cr_2O_7$ added per 4 Grms.	No. of c.c. $\frac{N}{100}$ $K_2Cr_2O_7$ recovered per 4 Grms.	No. of c.c. $\frac{N}{100}$ $K_2Cr_2O_7$ required for Titration of 100 c.c.	Blank.	Per Cent. of Chromium added.	Per Cent. of Chromium recovered.
	Per Cent.				C.c.		
Nickel ...	5.8	2.00	$0.35 \times 5 = 1.75$	0.70	0.35	0.0086	0.0076
Nickel ...	6.2	3.00	$0.60 \times 5 = 3.00$	0.95	0.35	0.0130	0.0130
Nickel ...	6.2	4.00	$0.85 \times 5 = 4.25$	1.20	0.35	0.0174	0.0184
Vanadium ...	1.0	2.00	$0.40 \times 5 = 2.00$	0.65	0.25	0.0086	0.0086
Tungsten ...	5.0	2.00	$0.37 \times 5 = 1.85$	0.62	0.25	0.0086	0.0077
Molybdenum	1.0	2.00	$0.37 \times 5 = 1.85$	0.62	0.25	0.0086	0.0077
Cobalt ...	5.0	2.00	$0.45 \times 5 = 2.25$	0.70	0.25	0.0086	0.0097

In the nickel and cobalt experiments sodium hydroxide was added after filtration, as described above.

In order to test the process more stringently, the tungstic acid which separated in the initial stages was not filtered off. The final filtrates from the vanadium, molybdenum, and tungsten experiments were tested for these metals, but none was found.

From these results it is evident that vanadium, molybdenum, and tungsten are completely removed by the ordinary process, and nickel and cobalt by the further treatment with sodium hydroxide.

The percentage of chromium present in a number of steels of high chromium content and varying composition was estimated both by the method under discussion and by the Vignal process, to see how the results would compare. The figures obtained are as follows:

Approximate Composition of Steel.				Chromium by Colorimetric Process.	Chromium by Vignal Process.
	Per Cent.			Per Cent.	Per Cent.
(a) Ni 0.5; W 15.8; Co 5.0; V 0.8				3.11	3.07
(b) Ni 2.8; Mo 0.5	...	...	..	0.82	0.85
(c) Ni 2.2	...	...	..	11.77	12.35
(d) Ni 2.4	...	..	..	1.50	1.55
(e) Ni 2.9	...	...	..	0.82	0.84
(f) Ni 2.2	...	...	...	1.83	1.80

These two series show very fair agreement when it is borne in mind that the colorimetric process is not particularly adapted to the estimation of large amounts of chromium, and that the tendency of the Vignal process is to give high results; the

figures, moreover, were obtained during the early stages of the process, and doubtless better agreement would be obtained now. To test the process to the utmost the full 4 grms. were taken in each case and the resulting deep-coloured solution diluted down. The last traces of nickel and cobalt were not removed with sodium hydroxide, the influence of the cobalt being evident in the high result of (a).

No correction has been made, throughout, for the volume occupied by the iron precipitate, the influence of which is well within the experimental error.

An attempt was made to get more delicate and concordant readings by adding potassium iodide to the acidified chromate filtrate and estimating the amount of iodine set free, but it was found that, at these dilutions, the decomposition of the hydriodic acid brought in a source of error considerably greater than that of the direct colorimetric process. Attempts were also made to complete the isolation of the chromium by precipitating it as lead or silver chromate; these, up to the present, have been unsuccessful, owing to the large amount of sulphate and phosphate present, whilst the mere presence of large amounts of alkali salts seem to prevent the precipitation of these small quantities of chromate.

The statement has been made that filter paper will reduce chromates either in acid or alkaline solution. In order to test whether the use of paper filters was having any influence, some of the results were repeated with the use of asbestos filters; the figures obtained were practically identical. It appears therefore that, under the conditions here set out, any error caused by reduction by the filter paper is negligible.

Another statement, which has been repeated in certain textbooks, i.e., that traces of chromium are not completely oxidised by permanganate in strongly acid solutions, caused a good deal of trouble in the early stages of the process, as it could not be determined whether the low results first obtained were due to incomplete oxidation or to removal of chromium as basic ferric chromate; even after the introduction of ammonium phosphate had produced a thoroughly reliable method it seemed doubtful whether its action was not merely to reduce the acidity. This point, which appeared to be of some importance, was tested in the following way: Traces of chromium (in the reduced form) were added to two 4-grm. samples of electrolytic iron, and the process was carried through in the usual way, except that the ammonium phosphate was added immediately before the introduction into the sodium hydroxide solution instead of before the oxidation. The following results were obtained:

No. of c.c. $\frac{N}{100}$ $K_2Cr_2O_7$ added.	No. of c.c. $\frac{N}{100}$ $K_2Cr_2O_7$ recovered.	No. of c.c. $\frac{N}{100}$ $K_2Cr_2O_7$ required for Titration of 100 c.c.	Blank.	Chromium added.	Chromium recovered.
2.00	$0.40 \times 5 = 2.00$	0.65	C.c.	Per Cent.	Per Cent.
4.00	$0.75 \times 5 = 3.75$	1.00	0.25	0.0087	0.0087
			0.25	0.0174	0.0163

From these results it appears that the chromium is completely oxidised in solutions of the acid strength here used. No reducing action caused by any manganese hydroxide, formed by the excess of manganese sulphate used, has been detected.

The action of the ammonium phosphate has not been fully explained. Some experiments were tried in which ammonium phosphate was replaced by sodium phosphate, but these gave very poor results; on the other hand, ammonium acetate was without any effect at all. It would seem that one function of the ammonium phosphate is to combine with the iron, preventing the formation of ferric hydroxide, which would drag down basic chromate.

In conclusion a few results may be cited, obtained by this process on samples of ordinary non-chromium steels:

Plain Carbon Steels. Chromium. Per Cent.	Nickel Steels. Chromium. Per Cent.
0.012	0.086
0.014	0.012
0.026	0.033
0.004	0.011
0.029	

+ + + + +

NOTES ON THE REACTIONS OF FULMINATE OF MERCURY WITH SODIUM THIOSULPHATE.

By F. H. DUPRÉ, M.B.E., AND P. V. DUPRÉ, M.B.E., A.C.G.I., F.I.C.

(*Read at the Meeting on December 1, 1920.*)

TITRATION.—During the war we had to examine large quantities of fulminate of mercury. The most rapid and accurate method seemed to be titration of the alkalinity in thiosulphate solution, but this involved the use of 3 grms. of potassium iodide for each titration—a rather expensive item with iodide at 18s. per lb. We therefore sought a substitute which, while being cheaper, would be equally effective in preventing the loss of alkalinity which occurs when the potassium iodide is omitted.

The problem was investigated from the point of view that if the alkalinity produced by the solution of the fulminate could be immediately neutralised, the secondary reaction causing the loss could not occur. A material, therefore, was looked for which could be mixed with the thiosulphate solution, and which, on the one hand, was capable of neutralising the alkali formed, and, on the other, would not act on the thiosulphate. Such materials as acetic acid, tartaric acid, oxalic acid, citric acid, salicylic acid, etc., all decompose thiosulphate. Boric acid was tried and found to be exactly what was wanted. It had the added advantage of having no action on methyl orange, and the titration could, therefore, be carried out exactly as when using iodide.

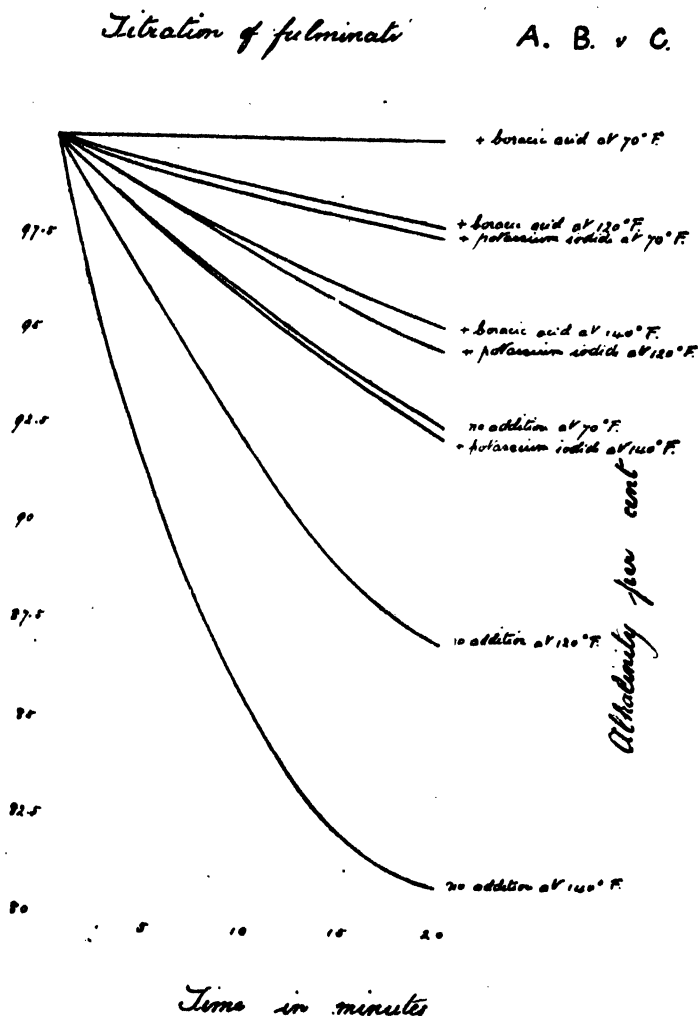
Comparative experiments were then made as follows:

- (1) 0.3 grms. fulminate, 25 c.c. 20 per cent. sodium thiosulphate solution.
- (2) 0.3 grms. fulminate, 25 c.c. 20 per cent. sodium thiosulphate, 3 grms. potassium iodide.

(3) 0.3 grms. fulminate, 25 c.c. 20 per cent. sodium thiosulphate solution, 1 gm. boric acid.

The results obtained at various temperatures are given in curves A, B, and C.

It will be seen that the boric acid is even more effective than potassium iodide, both at ordinary and raised temperatures. Since in the case of large crystal



fulminate its solution in thiosulphate takes some time, this effect of boric acid is very useful.

DESTRUCTION OF FULMINATE.—The next question which arose was connected with the destruction of waste fulminate. We had recommended the use of strong thiosulphate solution as being the quickest and safest method. It was suggested that the decomposition of the fulminate so caused was not permanent. In order to test this, a quantity of the solution of fulminate in thiosulphate solution was evaporated to

dryness and the residue examined. No trace of explosive property remained. In case this might be due to the heating, some of the solution was soaked up in filter paper and allowed to evaporate spontaneously at ordinary temperatures. Again it was found that not the faintest tendency to explosion remained. We therefore concluded that the decomposition was complete and permanent.* There are, however, two necessary precautions to be taken. Firstly, if the fulminate be simply put into the solution and allowed to settle to the bottom of the vessel, the complete destruction may take a very long time; efficient agitation of the liquid is therefore necessary; with such agitation the decomposition is rapidly completed. Secondly, an excess of thiosulphate is absolutely essential. If there be not sufficient thiosulphate present to destroy the fulminate completely, the cyanide produced in the reaction dissolves some unaltered fulminate, and this, on standing, crystallises out again. The danger involved in this is that, owing to the complete solution of the fulminate, it might be thought that the destruction was complete. The theoretical quantity of thiosulphate required is 1.746 grms. to each grm. of fulminate. We therefore recommended that a quantity of thiosulphate equal to three times the weight of fulminate to be destroyed should be used in the form of a 20 per. cent. solution.

A serious explosion which came to our notice occurred during the attempted destruction of detonators by boiling with thiosulphate solution. It is dangerous, in our opinion, to attempt to destroy detonators by treatment with thiosulphate, owing to the extreme slowness of the action, due partly to the compression of the fulminate, and partly to the protection from the solution by the shell, and, in some cases, covering metal discs or varnish, but it was thought desirable to try and discover the reason of the accident.

A considerable quantity of fulminate, about 25 grms., was put into the necessary amount of thiosulphate solution and vigorously stirred. The solution was completed in from a quarter to half a minute, and by taking the temperature before and after solution it was found that there was not the slightest rise in temperature, even when the solution was carried out at as high a temperature as 50° C. The solution which had been made at ordinary temperatures, however, showed on standing a slight rise in temperature.

Experiments were therefore made as follows: The solution was made as above, and the mixture immediately transferred to a thermos flask, the latter being closed by a cork through which a thermometer passed into the solution. The temperature was then taken at intervals extending over considerable periods, solution being made at various temperatures.

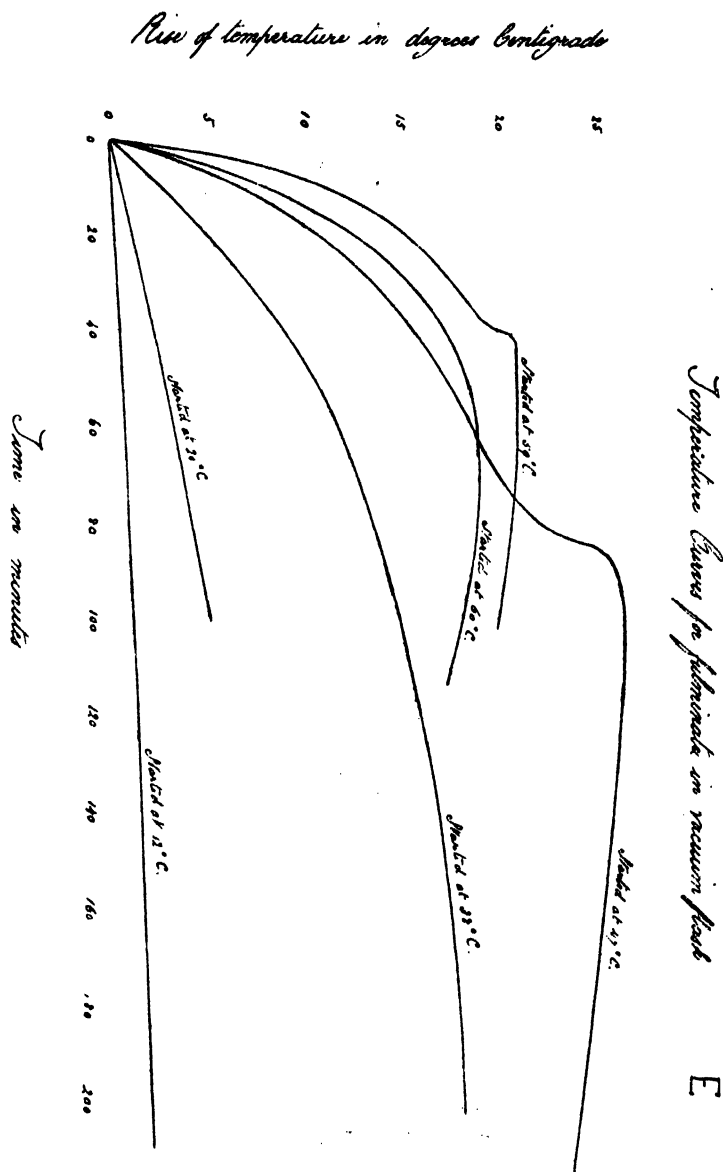
Curve E gives the results. It will be seen that in every case there is a marked rise in temperature, the more rapid the higher be the temperature at which the solution is made.

We therefore concluded that this might very possibly explain the accident. It seemed quite possible that sufficient heat might be generated by the decomposition of some of the fulminate to cause the ignition and explosion of the remaining unaltered fulminate.

In order to find the exact bearing the above effect had on our recommended

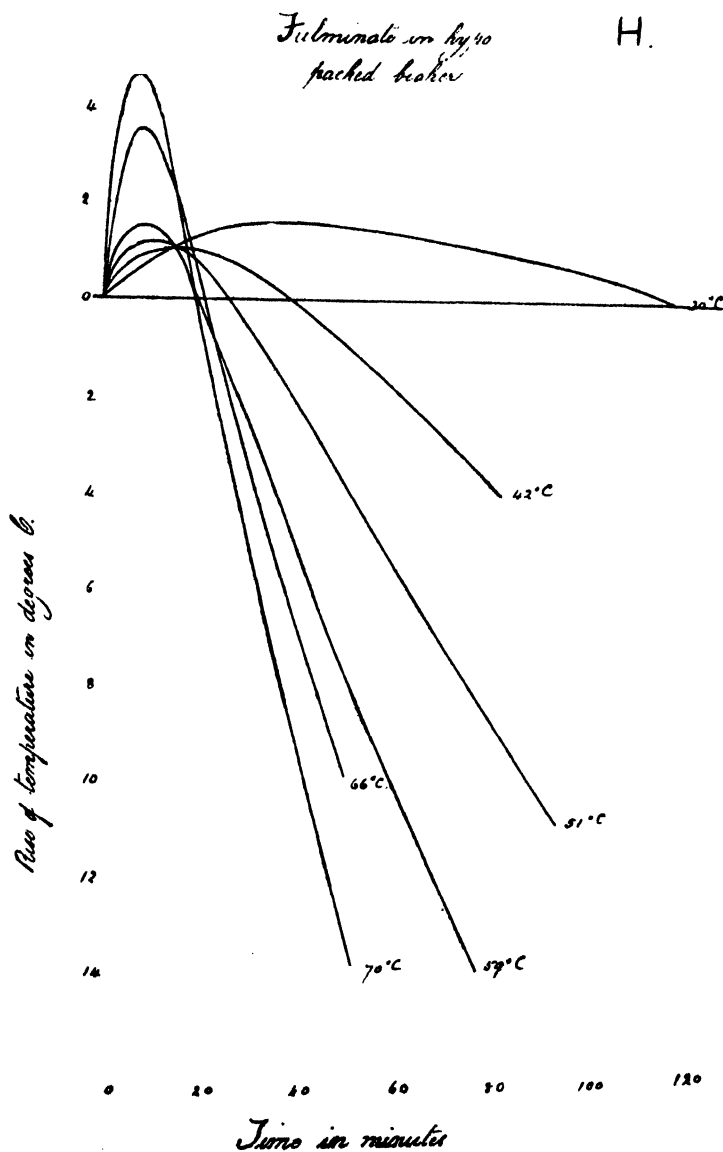
* The results of experiments described later confirm this.

method of destruction of loose fulminate, further experiments were made. Since the destruction was carried out in an open bucket the rate of loss of heat under such conditions from 100° C. downwards was determined. A beaker of a



convenient size for experiment was then packed in asbestos wool in such a way that the loss of heat occurred at as nearly as possible the rate found for the bucket, and the above experiment was repeated, replacing the thermos flask by the packed

beaker. It was then found that, even when solution was made at 70°C ., after a slight rise during the first few minutes, the loss of heat by radiation, etc., was more than the gain of heat due to chemical action. We therefore considered that it was



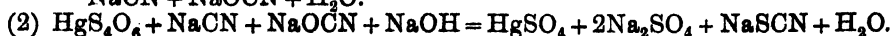
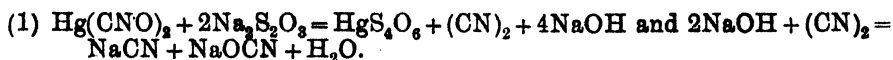
quite safe to continue the destruction of waste fulminate under the same conditions as before.*

Curve H gives the results of the experiments with fulminate.

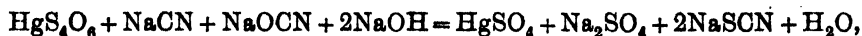
* Incidentally this, as mentioned above, confirms the supposition that the decomposition is non-reversible.

We then thought it would be of interest to try and follow the decompositions causing this rise of temperature.

There seems to be no entirely definite knowledge as to the reactions occurring during the solution of the fulminate or the ultimate product. Mr. Heaven, in a paper read before the Society of Chemical Industry (*J. Soc. Chem. Ind.*), gives Marshall's equation as follows :

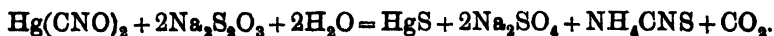


He points out that equation (2) is mathematically incorrect, and suggests that it can only be regarded as a statement in symbols. It is possible, however, that the mistake is one of printing, and that the equation should read :



which is, at any rate, mathematically correct.

As it was desired to ascertain the final products, the solution containing 2 equivalents of sodium thiosulphate to 1 of fulminate was boiled for a short time and then analysed. During the heating, a precipitate, sometimes red and sometimes black, was thrown down, and this proved to be sulphide of mercury; other radicles also present were sulphate and thiocyanate. Estimations were made of sulphide of mercury, sodium, SO_4 , and CNS, but these did not add up to the weight of fulminate and thiosulphate taken. A number of different experiments gave similar results. It had been noticed in the experiments with the thermos flask that when the temperature approached the maximum the solution appeared to boil. This proved to be due to the evolution of carbon dioxide, the amount of which was found to bring the sum of the products practically up to the weight of thiosulphate and fulminate taken; but still the equation could not be made to balance. It was then found that if the solution filtered from the mercury sulphide were evaporated to dryness, gently ignited, and weighed, it gave the same figure as when it was sulphated—i.e., the whole of the sodium present was there as sulphate. This meant that the thiocyanate present was not the sodium salt.* Ammonia was therefore tested for, and found to be present. Applying this to the solution of the equation we get



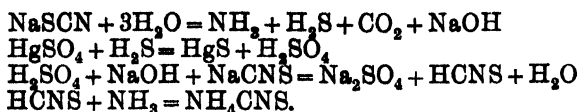
This is mathematically correct, and the analytical figures strongly support it. The following table gives the proportions of the different ingredients calculated from the equation and found by analysis, taking 100 grms. of fulminate and 174.6 grms. of sodium thiosulphate :

Substance.	Theory.	Found.
HgS	81.7	82.0
Na_2SO_4	100.0	100.0
NH_4CNS	26.8	26.6
CO_2	15.5	15.0

* This is not very conclusive, as the difference between the weight of sodium thiocyanate and its equivalent of sulphate is not very great.

This, therefore, is probably correct for the ultimate products, but it is very difficult to explain the presence of the ammonium thiocyanate if the second reaction given by Marshall is correct.

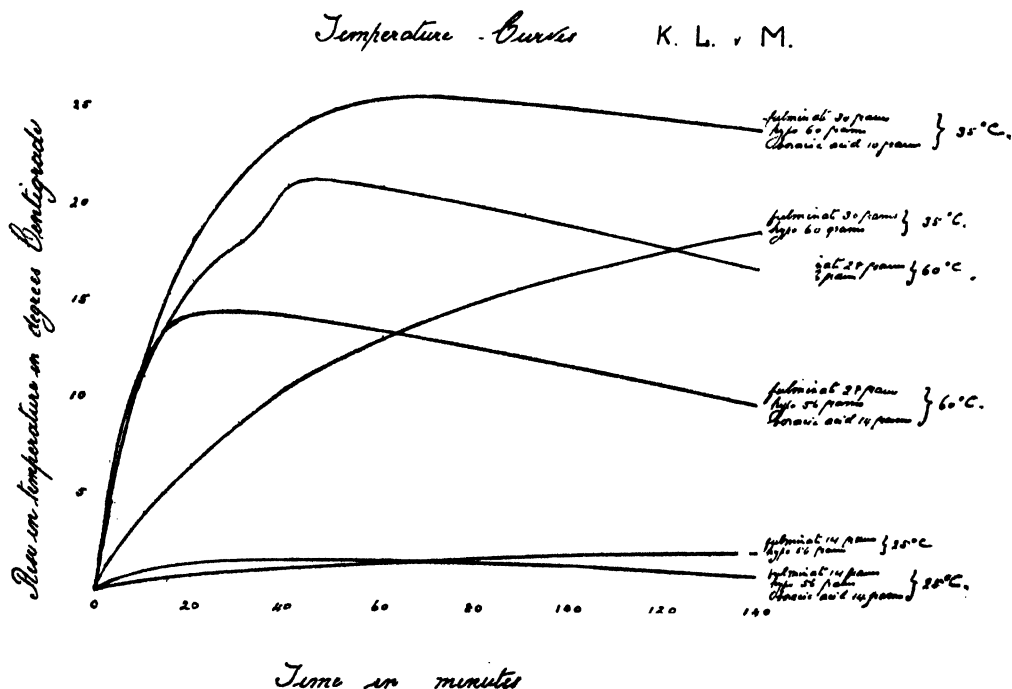
The following reactions are possible, and might explain the final result:



Collecting these we would get—



This would give the required result from Mr. Marshall's second equation, as amended.



EFFECT OF BORIC ACID.—Some experiments were then carried out to see whether the addition of boric acid to the solution in thiosulphate solution would prevent or delay the rise in temperature, since it did prevent the loss of alkalinity. The former experiments with the thermos flask were repeated with the addition of boric acid to the solution. The results are given in curves K, L, and M. It will be seen that the effect is somewhat unexpected, the addition of boric acid in the proportion of 1 part to 3 parts of fulminate considerably increases the rate of rise of temperature, the total rise being about the same as without addition. With 1 part boric acid to 2 parts fulminate the rate of rise is practically unaffected, but the total rise is

considerably diminished. With 1 part boric acid to 1 part fulminate the rate is increased for a short time, but the total rise is small. It must be remembered that in all the above curves the small loss of heat which occurs even with the thermos flask is ignored.

In the titration experiments the proportion of boric acid to fulminate was about 10 to 3; the above experiment was therefore repeated with the addition of a similar proportion of boric acid. It was then found that, allowing for the above-mentioned loss of heat, no change of temperature occurred for three hours. Parallel experiments to the above, in which small portions of the solution were titrated periodically, showed that whenever the temperature rose there was a corresponding loss of alkalinity, but in the last case no loss occurred in the three hours; in fact, after twenty-four hours the titration still gave the same figure. There was no sulphate nor thiocyanate present in this solution even after the twenty-four hours.

It thus appears that sufficient boric acid at least is required to form the usual sodium salt, the pyroborate or borax, $\text{Na}_2\text{B}_4\text{O}_7$. The proportions required, corresponding to the triborate, metaborate, and pyroborate, would be, 1 part fulminate, 0.29 parts boric acid; 1 part fulminate, 0.87 part boric acid, and 1 part fulminate, 1.75 parts boric acid respectively. Subsequent experiment has proved this to be the case.

* * * * *

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Comparison of Fat Tests in Milk as determined by a Milk-Testing Association and by a Creamery. H. C. Troy. (*Cornell Univ. Agric. Exper. Stat.*, Bull. 400, 1920, pp. 1-66.)—During recent years the practice of selling milk wholesale on a weight and fat-percentage basis has become general in New York State; the seller (farmer) is usually a member of an association which sends a tester once a month to examine the milk from each cow of the herd in the evening, and again the following morning. The mixed milk (not necessarily the whole supply of the herd) is delivered each morning at a receiving station, where a sample of the milk is placed in a composite sample bottle, and mercuric chloride is added as a preservative. Twice a month these composite samples are sent to the creamery and analysed. Results obtained during a year's working showed that when the fat was estimated once a month in two successive milkings the amount varied frequently by more than 0.5 per cent. from the preceding or following monthly test; the range of variation in the herd milk was less. The average fat content of the milk secreted at two successive milkings of a herd ranged from 0.2 to 0.3 per cent. above or below the fat percentage for the month, as found by the creamery, and differences of 0.5 per cent. for monthly periods were not unusual. During thirty-two complete

annual records, the differences between the two sets of tests were less than 0.1 per cent. in 59 per cent. of the records, and between 0.1 and 0.2 per cent. in 31 per cent. of the records. The daily fluctuations of the fat percentage in herd milk, and the association method of accepting the fat percentage in two successive milkings as the average for the month, did not ensure a close agreement between the results of the two tests for the monthly periods, but the association method permitted a fair estimate to be made of the fat content of the milk produced by a herd during a year.

W. P. S.

Comparison of Methods for the Estimation of the Fat Content of Skimmed Milk. T. J. McInerney and H. C. Troy. (*Cornell Univ. Agric. Exper. Stat.*, Bull. 401, 1920, pp. 1-85.)—Comparative tests with the Adams, Rose-Gottlieb, and Babcock methods for the estimation of fat in skimmed milk yielded results showing that the following modifications should be made in the Babcock method: At least 25 c.c. of sulphuric acid must be used, the temperature of the testing machine should be not less than 82°C., the centrifugal machine should have a disc at least 15 inches in diameter and be run at a minimum speed of 1,800 revolutions per minute, and the mixture should be centrifuged for ten, two, and one minute periods.

W. P. S.

Phosphorus in Butter. J. T. Cuisick. (*Cornell Univ. Agric. Exper. Stat.*, Memoir 30, 1920, pp. 159-186.)—In churning, about one-fourth of the total phosphorus of the cream is retained in the butter, the amount in the latter ranging from 0.025 to 0.041 per cent. of P_2O_5 . During storage, the soluble organic phosphorus compounds decompose, yielding inorganic phosphorus compounds, and the rate of this change depends upon the methods used in treating the milk and cream before churning. The presence of salt has a marked effect in causing decomposition of proteins in butter during storage, even at -10°C. The new alcohol-soluble protein discovered by Osborne and Wakemann (1918) in milk is also present in butter. Under certain conditions, bacteria are the controlling factors in the chemical changes of the phosphorus compounds of butter. The decomposition of lecithin and the formation of methylamine is the cause of fishy flavour in butter. When a fishy flavour develops in butter there is always an appreciable loss of soluble organic phosphorus.

W. P. S.

Lecithin Content of Butter and its possible Relationship to Fishy Flavour. G. C. Supplee. (*Cornell Univ. Agric. Exper. Stat.*, Memoir 29, 1920, pp. 101-150.)—Butter contains from 0.04 to 0.07 per cent. of lecithin, and there are indications that lecithin is the source of trimethylamine in butter, the amine giving a fishy flavour to the product. The presence of lactic acid and bacterial action are the controlling factors in the production of the trimethylamine. Certain butters, however, which possess a fishy flavour do not contain trimethylamine.

W. P. S.

Curua Palm Oil. (*Bull. Imperial Inst.*, 1920, 18, 172-174.)—Curua fruits (*Attalea spectabilis*), received from Brazil, were composed of pericarp, 15.5 per cent.,

shell, 71·3 per cent., and kernel, 13·2 per cent. The dry kernels yielded 65·3 per cent. of semi-solid greenish oil having the following characters :

Specific gravity at 100°/15° C.	...	...	0·8693
Refractive index at 40° C.	...	...	1·447
Melting-point	...	...	23·6° C.
Solidifying point of fatty acids	...	...	24·6° C.
Acid value	...	...	1·2
Saponification value	...	...	259·5
Iodine value	...	...	8·9
Unsaponifiable matter	...	...	0·36 per cent.
Reichert-Meissl value	...	...	equivalent to 6·26 c.c. of $\frac{N}{10}$ alkali per 5 grms. of fat
Polenske value	...	...	equivalent to 15·6 c.c. of $\frac{N}{10}$ alkali per 5 grms. of fat

W. P. S.

Cantaloup Seed Oil. W. F. Baughman, D. Brauns, and G. S. Jamieson. (*J. Amer. Chem. Soc.*, 1920, **42**, 2398-2401.)—Cantaloup (*Cucumis melo* L.) seeds yielded 30·4 per cent. of a pale yellow oil possessing a pleasant odour and taste, and having the following physical and chemical characters :

Specific gravity at 25°/25° C.	...	...	...	0·9210
Refractive index at 20° C.	...	...	...	1·4725
Iodine value (Hanus)	...	...	...	125·9
Saponification value	...	...	...	192·3
Reichert-Meissl value	...	...	...	0·33
Polenske value	...	...	...	0·26
Acetyl value	...	...	...	15·8
Acid value	...	...	...	0·43
Unsaponifiable matter	...	...	...	1·1 per cent.
Insoluble fatty acids	...	...	...	94·0 per cent.
Unsaturated fatty acids	...	...	...	80·2 per cent.
Saturated fatty acids	...	...	...	14·3 per cent.

The composition of the oil was: Myristin, 0·3; palmitin, 10·2; stearin, 4·5; olein, 27·2; linolin, 56·6; and unsaponifiable matter, 1·1 per cent.—Total, 99·9 per cent.

W. P. S.

The Otoba Nutmeg. (*Bull. Imperial Inst.*, 1920, **18**, 168-171.)—A forest tree (*Myristica otoba*), growing in the mountains of Columbia, produces spherical seeds 0·8 inch in diameter, consisting of shell 30 per cent. and kernel 70 per cent. On steam distillation the kernels yielded 7·2 per cent. of a colourless volatile oil, having the following characters :

Specific gravity at 15°/15° C.	...	...	0·894
Optical rotation	...	...	+ 79·550
Refractive index at 25° C.	...	...	1·502
Acid value	...	...	13·6
Ester value	...	...	Nil.
Ester value after acetylation	...	...	20·0
Solubility in 90 per cent. alcohol	...	...	Soluble in 16 vols. at 15° C.

The oil is deficient in certain odoriferous constituents present in ordinary nutmeg oil; it consists principally of terpenes, and cannot be used as a substitute for nutmeg oil. After the volatile oil had been removed, the dry kernels yielded 67.3 per cent. of fat somewhat resembling that obtained from nutmegs. The characters of this fat were:

Specific gravity at 100°/15° C.	...	...	0.892
Melting point	...	...	37.8° C.
Acid value	...	...	16.8
Saponification value	...	...	198.9
Iodine value	...	...	20.1
Titre test	...	...	37.2° C.

W. P. S.

Fractionation of Chaulmoogra Oil. A. L. Dean and R. Wrenshall. (*J. Amer. Chem. Soc.*, 1920, 42, 2626-2645.)—It is not possible to effect a satisfactory separation of the chaulmoogric and hydnocarpic acids in the mixed fatty acids of chaulmoogra oil by direct fractional crystallisation or fractional precipitation with barium acetate. Fractional distillation of the ethyl esters is less satisfactory than direct fractional distillation of the fatty acids *in vacuo*. By this means three fractions are obtained, in the first of which hydnocarpic acid predominates, whilst the third contains the main portion of the chaulmoogric acid. The intermediate fraction consists of mixed fatty acids, probably a eutectic mixture, which cannot be separated by fractional crystallisation. By adding this mixed fraction to a further portion of fatty acids for distillation the yields of hydnocarpic and chaulmoogric acids are increased in their respective fractions. The chaulmoogric acid fraction is recrystallized from 80 per cent. alcohol, 20 c.c. being used for each 5 grms., whilst the hydnocarpic acid fraction is treated in the same way, except that material melting above 35°C. is recrystallised from petroleum spirit (30 c.c. per 5 grms.). Even in the case of low grade chaulmoogric oil, at least 100 grms. of pure chaulmoogric acid (m.-pt. 68°C.; iodine value, 90.1; specific rotation + 56°), and at least 50 grms. of pure hydnocarpic acid (m.-pt. 59°C.; iodine value, 100.2; specific rotation + 68.1°) should be obtained.

"Taifushi" Oil. S. Keimatsu. (*J. Pharm. Soc. Japan*, 1920, 283-286.)—"Taifushi" oil, which is the chaulmoogra oil of the Japanese Pharmacopœia, is apparently derived from the seeds of *Hydnocarpus anthelmintica*, and not from those of *Taraktogenos Kurzii*, the source of genuine chaulmoogra oil. A sample of the Japanese product had the following characters: M.-pt., 22-23° C.; sp. gr. 0.952 at 15° C.; $[\alpha]_D = +51.2^\circ$; acid value, 12.06; saponification value, 20.30; and iodine value, 85.05. It consisted mainly of the glycerides of chaulmoogric and hydnocarpic acids. The latter acid, when oxidised at a low temperature, yielded a dihydroxy-hydnocarpic acid, $C_{15}H_{27}(OH)_2.COOH$, melting at 83° C.

Estimation of Methyl Alcohol in Cacao-Beans and Husks. C. H. Koers. (*Chem. Weekblad*, 1920, 17, 578-579.)—The total methyl alcohol was estimated in ten varieties of cacao-beans and five varieties of husks, with a view to the application

of this method as a means of determining the adulteration of cocoa with husks. The results were calculated free from fat and ash. In the beans the total methyl alcohol varied between 0.19 and 0.23 per cent., the results agreeing fairly closely with those obtained by von Fellenberg (*Mitt. Geb. Lebensmittundersuch. und Hyg.*). With the husks, however, much lower values were obtained in some cases than those recorded by von Fellenberg; the amounts found were 0.43–0.89 per cent. No safe conclusion as to adulteration with husks can be drawn from an estimation of the total methyl alcohol content of cocoa. Further, seven varieties of manufactured cocoa contained only 0.10–0.26 per cent. of methyl alcohol. These low figures are possibly due to loss of alcohol brought about by the action of potassium carbonate used in the treatment of the beans.

W. J. W.

Detection of Methyl Alcohol in Liqueurs and Spirits. L. Hoton. (*Ann. Falsif.*, 1920, 13, 490–491.)—The test proposed depends on the determination of the initial boiling-point of the liquid. Two hundred and fifty c.c. of the sample are distilled, with the aid of an efficient fractionating column provided with a thermometer at the top, and the temperature of the vapours is observed. With a 20 per cent. (by vol.) ethyl alcohol solution, the temperature of the distilling vapours, after the first thirty seconds, reaches 78° C., and remains constant for some minutes. If a small quantity of methyl alcohol be present, this temperature is diminished by several degrees.

W. P. S.

Essential Oil from the Berries of *Juniperus phoenicea* from Sardinia. E. Puxeddu and F. Vodret. (*Gazz. Chim. Ital.*, 1920, 50, ii., 245–257.)—These berries, when macerated in water for at least two days and then distilled, yield about 2.5 per cent. of an essential oil which is colourless and transparent, but turns slightly yellow in the light. It has a pronounced aromatic odour and yields a smell of turpentine when a drop is rubbed between the hands. It tastes like camphor and burns with a smoky flame and an odour of resin. Its constants were: Specific gravity, 0.8658 at 15°; $[\alpha]_D^{25} + 16.84^\circ$; viscosity, $\eta = 1.7259$ at 18.5°; solubility at 27°, nil in 70 or 80 per cent. alcohol, 1.16 in 90 per cent. alcohol; refractive index for daylight at 20°, 1.4675; reaction, neutral; saponification value, 14–15; acid value, 2.49; acetyl value, 35; aldehydes and phenols, absent; Maumené figure, 136.80; iodine value (Hübl), 285.53, varying results being obtained if the proportion of iodine employed is varied; bromine value, 264.55. Various colour reactions are described. Distillation under diminished pressure yielded a principal fraction, boiling at 74° under 30 mm. pressure, and having specific gravity 0.8543 at 25°, refractive index 1.4644, $[\alpha]_D^{25} + 23.94^\circ$.

T. H. P.

Estimation of Morphine in Opium. A. Jermsted. (*Ann. Chim. anal.*, 1920, 2, 354–358.)—A modification of the Helfenberg method is recommended, the procedure being essentially that prescribed in the Swiss Codex. Six grms. of the opium are ground to a paste with 6 grms. of water; the paste is rinsed into a weighed flask, and water is added to make the weight up to 54 grms. The mixture is shaken occasionally during thirty minutes, and then filtered; 40 grms. of the filtrate are treated

with 2.9 grms. of *N* ammonia and filtered; and 36 grms. of this filtrate are shaken in a flask with 7 grms. of ether and 4 grms. of *N* ammonia. After ten minutes, a further quantity of 10 c.c. of ether is added, and the mixture is placed aside for thirty minutes, after which the ethereal solution is poured through a filter, and the crystalline magma is washed three times with 5 c.c. of water saturated with ether. The filter and precipitation flask are dried at 100° C.; the portion of the crystals on the filter is returned as far as possible to the flask, dissolved in 25 c.c. of $\frac{N}{10}$ hydrochloric acid, the solution poured through the filter, and the latter and the flask washed with about 75 c.c. of water. The filtrate and washings are then treated with 5 drops of methyl-red solution, and the excess of acid is titrated with $\frac{N}{10}$ sodium hydroxide solution. Each c.c. of $\frac{N}{10}$ hydrochloric acid is equivalent to 0.0285 gram. of anhydrous morphine.

W. P. S.

Quantity of Ammonium Salts in Various Opiums. L. J. Thomson. (*Pharm. J.*, 1920, 105, 550.)—Opiums of different origin yielded the following percentage of quantities of ammonia (NH_3) when distilled with sodium carbonate solution:

	Highest.	Lowest.	Average.
Turkish (21 samples)	0.47	0.09	0.22
Indian (48 samples)	0.27	0.17	0.21
Persian (2 samples)	0.21	0.15	0.18

W. P. S.

New Reaction of Saccharin. L. Thevenon. (*J. Pharm. Chim.*, 1920, 22, 421-422.)—Diazotisation of saccharin and coupling of the resulting diazo-chloride with β -naphthol yields β -naphtholbenzoic acid, which is a red colouring matter. This reaction is carried out as follows: To 0.1 gram. of saccharin dissolved in 25 c.c. of water are added 10 c.c. of 0.1 per cent. sodium nitrite solution and 6 drops of dilute sulphuric acid (1:3), the liquid being shaken, allowed to stand for some minutes, and then treated with 0.1 gram. of β -naphthol. The red coloration thus developed is very intense and persistent, and is fixed by wool and silk. To detect saccharin in foodstuffs, the latter are extracted with water, alcohol, or other solvent, and the liquid extract mixed with a few drops of 1:3 sulphuric acid and treated as above.

T. H. P.

Yacca Gum. (*Chem. Trade J.*, 1920, 47, 803.)—The gum from *Xanthorrhoea hastilis* is soluble in alcohol and in aqueous solutions of the caustic alkalies, but is not dissolved by turpentine, benzene, linseed oil, or the usual hydrocarbon solvents. On addition of acid to the alkaline solutions the gum is more or less completely precipitated as a dull yellowish powder. Cold strong sulphuric acid partially dissolves the gum, forming a deep red solution, which, on the addition of water, gives a

brownish-red precipitate. When freed from sulphuric acid, this precipitate dissolves in water, and is reprecipitated on the addition of a little sulphuric acid to the solution.

T. J. W.

Adulteration of Gum Tragacanth with Gum Arabic. L. Thevenon. (*Ann. Falsif.*, 1920, 13, 489.)—Powdered gum tragacanth is sometimes adulterated with the cheaper gum arabic. The presence of the latter may be detected by treating a portion of the sample mixed with water with an equal volume of 4 per cent. pyramidon (dimethylaminophenyldimethylpyrazolone) solution, and then adding 10 drops of hydrogen peroxide (12 vol.). A blue-violet coloration develops within five to thirty minutes, according to the quantity of gum arabic present. The test will detect as little as 1 part of gum arabic in 20 parts of gum tragacanth.

W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Estimation of Phosphatides. D. H. Brauns and J. A. MacLaughlin. (*J. Amer. Chem. Soc.*, 1920, 42, 2238-2250.)—The method proposed involves the separation of the phosphatides from the other substances in the material to be analysed and the estimation of phosphorus, chlorine, and amino nitrogen in the mixed phosphatides. *Separation of Phosphatides.*—The material is extracted with hot alcohol, and the alcoholic extract is evaporated. Animal or vegetable tissues which cannot be reduced to a powder are dried previously with anhydrous sodium sulphate and calcium sulphate; liquids are treated similarly, whilst non-volatile liquid (e.g., glycerol) preparations are boiled directly with alcohol. The dry alcoholic extract is mixed with anhydrous sodium and calcium sulphate and extracted with ether, and the ethereal extract is evaporated to a volume of about 40 c.c., and washed with 15 c.c. of saturated sodium chloride solution (this removes traces of inorganic phosphates). The sodium chloride solution is drawn off, washed with 10 c.c. of ether, the united ethereal solutions are evaporated, and the residue dried and weighed. If possible, such a quantity of original material should have been taken as would yield not less than 0.2 gm. of mixed phosphatides. *Estimation of Phosphorus.*—The mixed phosphatides are dissolved in 100 c.c. of alcohol, and 5 c.c. of this solution are evaporated to dryness in a large test-tube; the residue obtained is then heated with a mixture of nitric and sulphuric acids (2 c.c. of each), and the heating continued until fumes of sulphuric acid appear. The mixture is cooled slightly, a few drops of dilute cane-sugar solution are added, the mixture again heated to expel the last traces of nitric acid, then rinsed into a basin with 10 c.c. of water, 10 c.c. of 30 per cent. ammonium nitrate solution are added, the whole heated to boiling and treated with 4 c.c. of 10 per cent. ammonium molybdate solution. The yellow precipitate is collected on a small asbestos filter, washed with ammonium nitrate solution, then transferred to a beaker by the aid of a small quantity of dilute sulphuric acid, and treated with 0.5 gm. of stannous chloride. After thirty minutes the blue solution is filtered through asbestos, the filtrate diluted to 100 c.c. with dilute sulphuric acid, and the coloration compared with that given by a known quantity of phosphorus under similar conditions. *Estimation of*

Choline and Amino Nitrogen.—About 0.3 grm. of the ether-soluble phosphatides, or an aliquot portion of the alcoholic solution after evaporation, is boiled with 30 c.c. of $\frac{N}{8}$ sulphuric acid under a reflux condenser for forty-eight hours; the solution is then cooled, filtered, the fatty acids are washed with water, and the filtrate is neutralised with barium hydroxide solution. The precipitate is collected on a filter, washed with hot water, and the filtrate acidified and evaporated to dryness. The dry residue is extracted with alcohol, the alcoholic solution concentrated to a volume of about 10 c.c., and alcoholic platinum chloride solution is added. After some time the precipitate is collected, dried at 105° C., and weighed. It consists of choline platino-chloride containing some amino-ethyl alcohol platinum chloride. The filtrate from this precipitate is acidified with hydrochloric acid, evaporated to dryness, the residue rendered slightly alkaline with sodium hydroxide, acidified with acetic acid, diluted to 2.5 c.c., and the amino nitrogen estimated by means of Van Slyke's micro-amino nitrogen apparatus. The amino nitrogen is also estimated in a portion of the precipitate. The weight of amino-ethyl alcohol platinum chloride is obtained by multiplying the weight of nitrogen by 19. The weight of the precipitate minus the weight of amino-ethyl alcohol platinum chloride equals the weight of pure choline platino-chloride; the latter, multiplied by 2.608 gives the weight of lecithin. The sum of the amino nitrogen in the filtrate and in the precipitate is multiplied by 53.20 to obtain the amount of kephalin.

W. P. S.

Distribution of Enzymes and Proteins in the Endosperm of the Wheat Berry. F. J. Martin. (*J. Soc. Chem. Ind.*, 1920, **39**, 327r.)—The enzymic activity of flour from various parts of the wheat berry was determined from the amount of carbon dioxide produced by a dough made from the flour, water, yeast, and a little salt, maintained at 29° C. for 24 hours. The results showed that the activity was highest in the portions close to the exterior of the endosperm, and gradually declined towards the centre. Portions of the same flours were used for the estimation of gluten, which varied from 13.8 per cent. in the centre to 21.6 per cent. in the outer portions. The consistency of the glutes obtained in the wet state was observed to vary considerably, and determinations made by measuring the volumes of the dough and the carbon dioxide evolved from doughs containing the same weight of gluten and consisting of flour, wheat starch, water, yeast, and salt, showed that the gluten from the centre of the berry possessed the highest gas-retaining capacity. The gas-retaining power is practically inversely proportional to the amount of gluten present, thus accounting for the uniform "strength" of flour from all parts of the berry. The above results were confirmed by baking tests, which showed that flour from all parts of the endosperm produced loaves obtaining the uniform value of 82 baker's marks.

T. J. W.

Presence of Hydrocyanic Acid in Linseed Cakes. Barishac. (*Ann. Falsif.*, 1920, **13**, 487-489.)—From the fact that the whole of the hydrocyanic acid known to be present in linseed cakes is obtained only when the material is distilled in the presence of a relatively large excess of hydrochloric acid, it is possible that the cyanogenetic glucoside may pass unchanged through the alimentary tract of an

animal. If this be the case, there seems to be little risk of poisoning when the cake is consumed. Linseed cake has been used for a very long time and in large quantity without any injurious effect having been noticed.*

W. P. S.

Presence of Hydrocyanic Acid in Linseed Cakes. E. Kohn-Abrest (*Ann. Falsif.*, 1920, 13, 482-487.)—The cyanogenetic glucoside occurring in linseed is concentrated in the pressed cake obtained when the oil is expressed, but the quantity in the cake appears to depend to some extent on the method of manufacture. Much of the hydrocyanic acid is lost during prolonged maceration of the seeds and by evaporation. Linseed grains of various origin (Indian, S. American, Russian, French, etc.) yielded about 0.02 per cent. of hydrocyanic acid; linseed cakes yielded up to 0.38 per cent. It is suggested that a maximum limit of 0.02 per cent. of hydrocyanic acid should be fixed for linseed cakes used for feeding animals.

W. P. S.

The Chemical Nature of Bacterial Anti-bodies. F. M. Huntoon, P. Masucci and E. Hannum. (*J. Amer. Chem. Soc.*, 1920, 42, 2654.)—By a series of indirect chemical, physical, and biological methods the authors arrive at the following conclusions: Anti-bodies are of colloidal nature, and the molecule contains a large number of atoms. They are unaffected by trypsin, not precipitated by solutions free from electrolytes, are insoluble in ether, unaffected by short exposure to 30 per cent. sodium chloride solution, by certain dilute acids and alkalies, and by temperatures up to 60° C. Above this temperature they are destroyed or altered in nature. From these results it is deduced that anti-bodies are not serum proteins, nor globulins, but may belong to the peptide group having a carboxyl-amino linkage.

T. J. W.

ORGANIC ANALYSIS.

New Method for the Separation of Highly Unsaturated Fatty Acids in Fish Oils. M. Tsujimoto. (*J. Chem. Ind., Tokyo*, 1920, 23 [272], reprint.)—The method is based upon the fact that the lithium salts of highly unsaturated fatty acids are soluble in acetone containing a little water (about 5 per cent. by volume), whereas the lithium salts of saturated and less unsaturated fatty acids are insoluble. About 5 grms. of the fatty acids from the fish oil are freed from unsaponifiable matter and dissolved in 20 c.c. of anhydrous acetone, and the solution neutralised with 4*N*-aqueous lithium hydroxide solution (phenolphthalein as indicator), the number of c.c. required being termed *a*. Then (6 - *a*) c.c. of water and 75 c.c. of acetone are added, and the flask corked and chilled in ice for two hours, after which the solution is filtered through dry filter paper placed in a funnel surrounded by ice. The filtrate is allowed to stand until its temperature has risen to that of the room; 50 c.c. are then evaporated, and the residue of soluble lithium salts decomposed with dilute hydrochloric acid. The liberated acids are dissolved in ether, the solution

* According to some authorities this depends largely upon the age of the animal.—EDITOR.

dried with anhydrous sodium sulphate and evaporated, and the residual acids weighed. The following results are typical of those obtained by this method:

	Original Fatty Acids (Free from Unsapoifiable Matter).			Highly Unsaturated Fatty Acids.				Acids Free from Highly Unsaturated Acids.	
	Iodine Value.	Neutralisa- tion Value.	Poly- bromides.	Yield.	Iodine Value.	Neutralisa- tion Value.	Refractive Index, 20° C.	Iodine Value.	Neutralisa- tion Value.
			Per Cent.	Per Cent.					
Japanese sardine oil ...	171	187	49	29.9	357	—	1.4965	83	191
Herring oil ...	108	193	15	15.0	203	168	1.4890	73	197
Codliver oil ...	169	190	40	29.2	360	172	1.4927	91	196
Sunfish liver oil ...	164	192	43	30.8	338	174	1.4910	91	194
Whale oil ...	114	—	22	13.6	306	177	1.4887	69	196

Vegetable oils and terrestrial animal oils gave only relatively small yields of highly unsaturated fatty acids—*e.g.*, 1.2 per cent. in the case of sesamé oil and 9.3 per cent. in the case of linseed oil. The acids thus separated, however, still contain a certain proportion of solid and less unsaturated acids, but the separation may be completed by fractional distillation of the methyl esters of the fatty acids. Further investigation has shown that the clupanodonic acid, first isolated by the author from Japanese sardine oil, is not a definite chemical compound, and that its formula $C_{18}H_{28}O_2$ ($C_nH_{2n-8}O_2$ series) is also incorrect. The principal highly unsaturated fatty acids in this oil appear to be $C_{20}H_{30}O_2$ and $C_{22}H_{34}O_2$ ($C_nH_{2n-10}O_2$ series), and it is suggested that the formula for clupanodonic acid should be changed to $C_{22}H_{31}O_2$, which forms the bromide $C_{22}H_{31}O_2Br_{10}$, containing 70.76 per cent. of bromine.

Estimation of Dextrose and Starch. F. A. Quisumbing. (*Philippine J. Science*, 1920, 16, 581-599.)—The estimation of starch in flours and other starch products is carried out as follows: Two to 3 grms. of the sample are well washed with 250 c.c. of cold water, and gelatinised by heating to boiling with about 150 c.c. of water, cooled to 40° C., and 25 c.c. of saliva, exactly neutralised with dilute hydrochloric acid, are run in, the temperature being maintained at 40° C. until the addition of iodine to a few drops yields no blue colour. The solution is filtered and diluted to 200 c.c., 15 c.c. of sulphuric acid run in, and the mixture boiled for three or four hours under a reflux condenser. On cooling, the solution is neutralised with sodium hydroxide solution and diluted to 500 c.c. Twenty-five c.c. of this solution, 50 c.c. of $\frac{N}{10}$ potassium permanganate solution, and 25 c.c. of 0.848 per cent. sodium carbonate solution, are mixed, and so heated that the temperature rises from 29° to 95° C. during a period of two minutes, and the latter temperature is kept constant for a further two minutes, after which 25 c.c. of 28 per cent. sulphuric acid and 25 c.c. of $\frac{N}{10}$ oxalic acid solution are run in, and the excess of oxalic acid titrated with $\frac{N}{10}$ potassium permanganate solution. The difference between the volumes of $\frac{N}{10}$ potassium permanganate solution used, and the $\frac{N}{10}$ oxalic acid added, represents the potassium permanganate used in oxidising the dextrose, and, by reference to a

table given in the original paper, the amount of starch present in the sample taken is found. When dealing with pure starches the preliminary gelatinisation and salivary digestion are omitted, whilst dextrose requires only the heating with alkaline permanganate and the subsequent titration. The method gives results showing excellent agreement with those obtained by Munson and Walker's modification of Fehling's method (*J. Assoc. Off. Agric. Chemists*, 1916, 1 and 2). Brief references are made to the work of earlier experimenters upon the action of alkalies and oxidising substances upon sugars, and preliminary experiments made during the evolution of the above method are described in detail. T. J. W.

The Individuality of Erythrodextrin. J. C. Blake. (*J. Amer. Chem. Soc.*, 1920, 42, 2673-2678.)—An erythrodextrin was isolated from five samples of dextrin (prepared by different methods from wheat and maizestarch), by the following series of operations. Fractional precipitation by alcohol of concentrations between 50 and 65 per cent., precipitation by ammonium sulphate, heating to 60° C. with slight excess of barium carbonate for two hours, precipitation by the addition of 42 per cent. alcohol and drying over calcium chloride to constant weight. The results of the operations were controlled by the determination on a 5 per cent. solution of the specific rotation at 25° C., the coloration produced by the addition of iodine, the time required for digestion to the achromic point (iodine) with saliva diluted 1 to 18, and the "glucose value" obtained with Benedict's reagent. The product obtained yielded the following values: Polarimetric reading +16.2; time required to reach achromic point, 2.25 minutes; and "glucose value," 1.6. The colour produced by iodine with erythrodextrin is at a maximum when the former is in slight excess, and a correction for the red colour due to this excess may be made by deducting from the red colour 40 per cent. of the total yellow colour observed. The presence of amylopectin has no disturbing influence, since the blue colour produced neutralises the yellow colour of the iodine, until excess of the latter is present. The erythrodextrin obtained by the above method is of great use in the estimation of relative amylolytic activity.

T. J. W.

Petrols containing Dissolved Gases. F. Bordas. (*Ann. Falsif.*, 1920, 13, 539-543.)—Certain motor spirits (petrol) sold recently in France have consisted of a "heavier" petroleum fraction containing an appreciable quantity (up to 10 per cent.) of dissolved gases, such as methane, ethane, propane, and butane. The quantity of gaseous constituents may be estimated by placing 200 c.c. of the sample in a flask attached to a reflux condenser, the top of the latter being connected with a tube which extends into a graduated cylinder containing 50 c.c. of ordinary kerosene; the cylinder is surrounded by a freezing mixture. The flask is heated to nearly the boiling-point of its contents for forty-five minutes, and the increase in volume of the kerosene then noted, after the latter has been brought to its original temperature. The difference in the specific gravity of a petrol before and after the removal of gaseous constituents is of the order of 0.010.

W. P. S.

Melting-Point of Coal. G. Charpy and J. Durand (*Comptes rend.*, 1920, 171, 1358-1360.)—The finely-ground coal is moistened with water, moulded into small

cylinders about 35 mm. in length, dried at 105° C., and sealed in glass tubes from which the air has been displaced by a prolonged current of hydrogen. The tube is placed in a copper envelope and heated in a lead bath, the temperature being measured by a mercury thermometer up to 460° C., and by a thermo-electric couple beyond that. The temperature is gradually raised, the tube being removed at intervals for inspection. The coal at first contracts, then apparently expands, and afterwards undergoes a second contraction characteristic of true conversion into coke. It is at the temperature corresponding with the maximum of the first contraction that fusion occurs, this being characterised by softening of the coal, which fills the glass tube completely. Of eight coals examined, six melted between 395° C. and 550° C., and the other two did not melt below 600° C. The fusibility is independent of the content of volatile matter, but, like the tendency to form coke, is directly connected with the state of oxidation; coals oxidised by heating at 120° C. no longer melt.

T. H. P.

Estimation of Phenols in Sheep Dips. K. B. Edwards and G. A. Freak. (*J. Soc. Chem. Ind.*, 1920, **39**, 326-327T.)—The method adopted by the U.S. Government consists in distilling the acidified dip with steam, dissolving the phenols in benzene, and shaking this solution with sodium hydroxide solution (1 : 3) in a graduated tube. After complete separation the increase in volume of the alkaline solution is measured, and each c.c. of the increase is assumed to represent 1 gm. of phenols. Investigation of this method by the authors has shown that in the case of pure phenols the results obtained vary within wide limits according to the nature of the phenol and its boiling-point. Thus phenol shows an apparent percentage of 92.5, whilst *p*-cresol yields a corresponding value of 102.5. Various fractions obtained by the distillation of commercial cresylic acid yielded similar results, that boiling below 195° C. showing an apparent percentage of 95, and the last fraction, boiling above 250° C., giving 117.5; whilst intermediate fractions showed a gradual rise in the percentage with the increase in the boiling-point. Since phenols, boiling above 250° C., are volatile with great difficulty when distilled with steam, the method is applicable only to those having a lower boiling-point, and any others present in the dips are omitted from the final estimation. Reference is made to a case where the specification required that 60 to 70 per cent. of the cresylic acid supplied should boil between 185° and 195° C. Tested by the method described, this sample showed 92 per cent. of phenols, whereas the actual amount present was 97 per cent.

T. J. W.

Oxidation of the Grignard Reagent. C. W. Porter and C. Steel. (*J. Amer. Chem. Soc.*, 1920, **42**, 2650-2654.)—The authors have studied the reactions of phenyl magnesium bromide by bubbling pure oxygen through an ethereal solution of the compound, and hydrolysing the resulting mixture with ice and dilute hydrochloric acid, the final products being identified and estimated. The yield of phenyl bromide is practically constant, indicating that this compound is formed by the direct action of magnesium upon phenyl bromide during the preparation of the Grignard reagent. The optimum temperature for the production of phenol is 0° C.,

when a yield of 22.9 per cent. is obtained. The presence of certain compounds and the increase in the yield of phenol when hydrolysis takes place in the presence of a reducing agent suggest the presence of phenyl peroxide as an intermediate product.

T. J. W.

Detection of Nitroso Compounds and Estimation of Nitro Derivatives in Smokeless Powder. C. Lutri. (*Giorn. Chim. Ind. Appl.*, 1920, 2, 557-559.)—Nitrous acid, nitrosamines, and nitroso-derivatives give an intense violet coloration with a 1 per cent. solution of α -naphthylamine in acetic acid. A similar coloration is also given by potassium chlorate, hydrogen peroxide, and lead peroxide, but not by ammonium persulphate or potassium nitrate. The presence of dinitrotoluene does not interfere with the application of the test to smokeless powders. *Estimation of Aromatic Nitro Compounds.*—Three grms. of the finely-divided explosive are extracted for about six hours with ether, the extract evaporated at a low temperature, and the residue heated on the water-bath beneath a reflux condenser for about six hours with 50 c.c. of 20 per cent. sulphuric acid, the flask being meanwhile frequently shaken. The liquid is then filtered, the filtrate extracted with benzene, the extract evaporated at about 50° C., and the residue weighed.

Estimation of Mineral Residue in Smokeless Powders. G. Lutri. (*Giorn. Chim. Ind. Appl.*, 1920, 2, 244.)—A method of estimating mineral residues or graphite in smokeless powders is based upon the solution of the organic constituents in pyridine: Five grms. of the powder in a fine state of division are mixed with about 8 grms. of pyridine, and the beaker immersed at intervals in a hot-water bath, until, after forty-eight hours, the viscid mass has become liquid. The contents of the beaker are then diluted with 20 c.c. of alcohol and filtered through a weighed Gooch crucible, and the residue washed successively with alcohol, ether, hot benzene, dilute hydrochloric acid, and water. Finally it is ignited until it shows signs of incandescence, cooled, and weighed.

Inks: Their Composition, Manufacture, and Methods of Testing. (*Circular No. 95, Bureau of Standards, Department of Commerce, U.S.A., 1920, pp. 24.*)—*U.S.A. Standard for Iron Writing Inks:* The U.S.A. Government has adopted the following specification for inks: The ink must be a gallotannate of iron ink, not inferior in any essential quality to one properly prepared according to the following formula: Tannic acid, 23.4 grms.; gallic acid, 7.7 grms.; ferrous sulphate, 30 grms.; dilute hydrochloric acid, U.S.P., 25 grms.; phenol, 1 gm.; and suitable blue dye, 2.2 grms., with water to make 1,000 c.c. at 15.6° C. *Duplicating inks* consist of a mixture of pigment (carbon, ferricyanides and ferrocyanides of iron, artificial ultramarine) and oil, such as castor-oil, sulphonated castor-oil, rosin oil, cotton-seed oil, sulphonated cotton-seed oil, and mineral oil. Some also contain rosin soap. **ANALYSIS OF WRITING INKS**—*Total Solids:* Ten grms. are evaporated to dryness, and the residue dried for one hour at 105° C. *Ash:* The residue is ignited at a low red heat. *Iron:* The ash is dissolved in 25 c.c. of hydrochloric acid, the solution diluted to 400 c.c., and the iron precipitated with ammonium

hydroxide and estimated gravimetrically. *Sulphuric Anhydride*: Ten grms. of the ink are evaporated in a silica dish, and the residue heated for an hour at 120°C ., and then ignited and cooled, and the dish heated in water for an hour on the water-bath. The solution is filtered, heated for thirty minutes on the water-bath with 5 c.c. of bromine water, acidified with hydrochloric acid, and treated with 10 c.c. of 10 per cent. barium chloride solution. The barium sulphate is filtered off after twelve hours. *Tannin*: Ten grms. of ink are mixed with 10 c.c. of concentrated hydrochloric acid in Kempf's extraction apparatus, and water added to about 2 inches below the overflow tube. The bulb is then filled with ethyl acetate, and the apparatus shaken until all tannin has been extracted (about two hours). The ethyl acetate extract is washed three times with a semi-saturated solution of potassium chloride to remove any iron salts present, and then evaporated *in vacuo*, the residue taken up with a little water, the solution filtered if necessary, and evaporated in a weighed dish, and the residue dried at 105°C . and weighed. *Chromium*: Chromium, which is usually present in logwood inks, is estimated by fusing the ash with 10 grms. of sodium carbonate in an oxidising atmosphere; the fused mass is treated with water, and filtered, the filtrate acidified with acetic acid, heated to boiling, and treated with 10 per cent. barium chloride. In the presence of chromium a yellow precipitate will be obtained. It is collected in a Gooch crucible, ignited, cooled, and weighed as Cr_2O_3 . *Specific Gravity*: This is determined at 15.6°C ., and compared with water at the same temperature. *Streak Tests*: The ink is applied to pieces of all-rag writing-paper of good quality, 11 by $5\frac{3}{4}$ inches, which are clamped on to a glass plate 8 inches wide, inclined at an angle of 45° , pipettes with a capacity of about 0.6 c.c. being used for the purpose. Regular streaks should be obtained, the head being oval, and the remainder of nearly uniform width. In the case of a very fluid ink the head is tapering, and the streak shows rapid contraction. *Resistance to Light and Reagents*: After the ink has become oxidised within the fibres of the paper (which will take from five to eight days) the streaks are cut laterally into four strips. One of these is immersed in water at the ordinary temperature, and a second exposed to ultraviolet light (or to direct sunlight), whilst the third and fourth are kept in the dark. Strip No. 4 is subsequently cut into small pieces and tested with various reagents, such as 95 per cent. alcohol; 90 parts by volume of water with 10 of ammonium hydroxide; 2 per cent. hydrochloric acid; 2 per cent. sodium hydroxide solution; and bleaching powder solution containing $\frac{N}{2000}$ available chlorine. The tests are relative, and the results should be compared with those given by the standard ink. **ANALYSIS OF CANCELLING INKS**—*Substances Volatile at the Ordinary Temperature*: A weighed quantity (about 5 grms. of the ink) is exposed to the air, and weighed after eighteen hours, and then after each day. The percentage loss in weight ought not to exceed 15 per cent. during the first seven days. *Penetration Test*: Strips of white blotting-paper, about 12 mm. wide and 25 cm. long, are cut from the same sheet, and introduced into 5 c.c. of the ink contained in a homoeopathic "shell vial" about 8 cm. long and 2 cm. in diameter. Portions of 5 c.c. of water are placed in two similar vials, and strips of blotting-paper also introduced into these. In each case the blotting-paper should be nearly vertical, and care should be taken that the liquid does not ascend by capillarity between the edges of

the strips and the sides of the vials. Exactly fifteen minutes from the time of insertion the height to which the liquid has ascended the strip of paper is measured by means of dividers and a millimetre rule, and a second set of readings is made after forty-five minutes. In each case the readings are expressed in terms of the penetrating power of water represented as 100, by dividing each result by the average of the results obtained with water, and multiplying the quotient by 100. The portions of the immersed strips may also be cut off, and extracted successively with petroleum spirit, alcohol, etc., to separate the constituents of the ink. The extent to which the dyes or lampblack have penetrated the paper affords an index of the quality of the ink. *Sedimentation Test*: The ink is introduced into a graduated 200 c.c. cylinder to the depth of 16 mm., and treated with a suitable solvent (alcohol, ether, benzene, petroleum spirit, etc.) so as to give 200 c.c. of solution. The cylinder is closed with a stopper and allowed to stand, and the layer of sediment measured, at intervals of fifteen minutes during the first hour, then each hour for several hours successively, and subsequently twice a day for a week to ten days. After settling is complete the height of the sedimentation should not equal or exceed 16 mm. The rate of sedimentation affords an index of the state of division of the carbon. In the case of some inks no appreciable layer is formed after ten days. *Lampblack*: Five grms. of the ink are diluted with a suitable solvent, and the deposited carbon collected in a Gooch crucible, washed with solvent, dried, and weighed, and the weight corrected for the amount of ash. *Glucose*: Three grms. of the ink are diluted with water, the dyestuffs precipitated with sodium hydroxide, the liquid filtered, and the sugars estimated in the filtrate by means of Fehling's solution.

Classification and Identification of Coal Tar Dyes. *Circulaire du Chef du Service des Finances et MM. les Chimistes en Chef des Laboratoires.* (*Ann. Falsif.*, 1920, 13, 193-456.)—Methods are described for separating the dyes into groups with a view to their subsequent identification, and suitable tests are described. An alphabetical list of the dyes is also given. The paper is unsuitable for abstraction, and the original should be consulted. W. P. S.

INORGANIC ANALYSIS.

Conductimetric Titration. J. M. Kolthoff. (*Chem. Weekblad*, 1920, 17, 694-701.)—The conditions under which the conductimetric method is applicable to the titration of strong and weak acids and bases have been investigated. Phenol, boric acid, and hydrogen cyanide give good results, and the method is also suitable for the estimation of aniline and its derivatives and alkaloids. Formic, oxalic, and tartaric acid may be titrated with ammonia, but boric acid is too weak. With mixtures of acids it is sometimes advantageous to add alcohol in order to reduce the dissociation constant. With boric acid and acetic acid no definite break in the curve is obtained, but with oxalic and tartaric acid, boric acid may be satisfactorily titrated. Phenol behaves similarly. Hydrogen cyanide may be titrated in presence of acetic acid, but cannot be determined with oxalic acid. The author gives many more examples of the applicability of the method. W. J. W.

Table of Highly Sensitive Rays of the Elements Applicable to Analytical Investigations. A. de Gramont. (*Comptes rend.*, 1920, 171, 1106-1109.)—The author gives for most of the elements the wave-lengths of the rays of great sensibility obtained in the condensed spark, either by direct observation on metals, alloys, and conducting minerals, or by fusion of non-conducting minerals or pulverulent precipitates with salts of the alkali metals. T. H. P.

Gas-Volumetric Estimation of Ammoniacal, Nitric, and Organic Nitrogen in Mixed Fertilisers. P. Baldi. (*Giorn. Chim. Ind. Appl.*, 1920, 2, 376-378.)—The fertiliser (10 grms.) is extracted with hot water, and the filtered extract and washings diluted to 200 c.c. Ten c.c. of the solution are used for the estimation of ammoniacal nitrogen by the hypobromite method, whilst nitric nitrogen is estimated by the same method in 50 c.c. of the solution, after reduction with Devarda's alloy, in presence of sodium hydroxide. Organic nitrogen in the solution and in the residue from the extraction is estimated by Kjeldahl's process, the ammonia being determined by the hypobromite method. The tabulated results show that the gas-volumetric method, applied as described, gives slightly lower proportions of nitrogen than those obtained by distillation of the ammonia.

Iodimetric Estimation of Amino Nitrogen. H. H. Willard and W. E. Cake. (*J. Amer. Chem. Soc.*, 1920, 42, 2646-2650.)—Material containing 10 or 15 mgrms. of nitrogen is weighed and heated with 15 c.c. of concentrated sulphuric acid for about ten minutes, or until the organic matter is completely charred and free evolution of sulphur dioxide has ceased. After cooling, solid potassium persulphate, weighing ten times the amount of the material taken, is added by means of a thistle tube, the mixture being gently warmed and then swirled until colourless, boiled for five minutes to decompose any excess of persulphate, diluted with 70 c.c. of water, and again boiled to remove any residual sulphur dioxide. After cooling, a few drops of methyl orange are added, and the solution is neutralised by the addition of 40 per cent. sodium hydroxide solution, the solution being frequently cooled in a stream of water to avoid loss of ammonia. Two drops of the sodium hydroxide solution are run in, followed by 10 c.c. of 0.6N sodium hypobromite, and the mixture allowed to stand for five minutes, after which 5 grms. of potassium iodide and 6 c.c. of hydrochloric acid are added, and the liberated iodine titrated with $\frac{N}{8}$ sodium thiosulphate solution. The sodium hypobromite solution is prepared by slowly adding 50 grms. of bromine to a well cooled solution of 30 grms. of sodium hydroxide in 800 c.c. of water. If kept in the dark at a temperature below 5° C. the solution retains its composition unaltered for a week, but at ordinary temperatures it should be standardised daily against pure ammonium sulphate or sodium thiosulphate. One c.c. of the 0.6N solution is equivalent to 2.8 mgrms. of nitrogen. Since nearly all samples of potassium phosphate contain ammonium salts, a blank determination should be made, and the result deducted from the results of any determinations. If iron salts be present, the addition of phosphoric acid will prevent the liberation of iodine due to these compounds. The method is more rapid than the usual Kjeldahl determination, and yields equally accurate results. T. J. W.

Estimation of Nitrite in Water. P. A. Meerburg. (*Chem. Weekblad*, 1920, 17, 577-578.)—The colorimetric reaction of nitrites in water with the Griess-Romijn reagent (naphthylamine, 1 part; sulphanilic acid, 10 parts; tartaric acid, 89 parts) is accelerated by the presence of sodium chloride, but retarded by bicarbonates. The latter salts should therefore first be neutralised with sulphuric acid, and 0.1 per cent. of sodium chloride should then be added both to the reagent and to the water under examination, after which the reaction will take place in thirty minutes.

W. J. W.

Rapid Method of Disintegrating Metal Alloys not attacked by Acids.
I. Compagno. (*Giorn. Chim. Ind. Appl.*, 1920, 2, 554-555.)—Metal alloys, such as ferrotungsten, ferrosilicon, etc., which are not attacked by mineral acids, may be rapidly disintegrated by fusing 0.25 to 0.5 gm. with a mixture of 52 per cent. of sodium carbonate, 20 per cent. of potassium nitrate, and 28 per cent. of coarse magnesium powder which has been dried for two hours at 100° C., and kept in a bottle with a tightly-fitting stopper. About 4 grms. of this mixture is placed in a nickel crucible, then the powdered alloy mixed with 2 grms. of the fusion reagent, and then 10 to 12 grms. of the latter, and finally 1 gm. of magnesium powder. After fusion the mass is cooled and treated with a mineral acid, and the solution boiled until gas is no longer evolved, and then analysed by the usual methods.

General Method for the Detection and Estimation of Arsenic. Kohn-Abrest. (*Comptes rend.*, 1920, 171, 1179-1182.)—Arsenic may be detected and estimated in any material, such as animal organs or liquids, foods, etc., in the following manner: One hundred grms. of the substance, chopped fine if necessary, are mixed in a flat-bottomed porcelain dish with 35 c.c. of a solution containing 200 grms. of crystalline magnesium nitrate per litre, and with 1 gm. of calcined magnesia. The basin is then heated to about 250° C. in an oven placed on a sand-bath. When the mass is dried and roasted, this usually requiring about three hours, the carbonaceous residue is ground in a mortar, returned to the basin, and introduced into a muffle furnace, the bottom of which is heated to dull redness (500° C.). After about two hours the ash, which is almost free from carbon, is mixed with 30 c.c. of dilute sulphuric acid (10 vols. per 100), which usually dissolves it completely. The liquid is diluted to 60 c.c., and the arsenic estimated by Marsh's method. When the percentage of arsenic in the organs to be examined lies between 0.04 and 0.00002, 70 to 80 per cent. of the total arsenic is obtained by this procedure. In the case of organic arsenic compounds, 0.15 to 0.2 gm. of the product is mixed in a 40 c.c. porcelain crucible with about 0.2 gm. of calcined magnesia and 6 c.c. of the 20 per cent. crystallised magnesium nitrate solution. The open crucible is kept in an oven at 110° C. until the mass is dry, and is then fitted with a flat-rimmed lid large enough to project about 5 mm. beyond the wall of the crucible, and placed in a muffle furnace heated to bright redness. After a few minutes the organic matter is burnt, the magnesium nitrate decomposed, and the arsenic converted into magnesium pyroarsenate. The ash remaining is dissolved in 30 c.c. of a hydrochloric acid solution containing 150 c.c. of acid of density 1.17 per litre, the solution being left

on a water-bath for five minutes, treated with 7.5 grms. of potassium iodide, and kept near the boiling-point for five minutes. The liquid is then cooled under the tap, and a rough indication of the arsenic obtained by titrating the free iodine with thiosulphate. The solution is afterwards rendered alkaline by the addition of sodium bicarbonate, and the arsenious compound titrated with iodine solution: 1 c.c. of $\frac{N}{10}$ iodine solution corresponds with 0.00375 gm. of arsenic. In most cases these two titrations give concordant results; but in one case in which the combustion proved difficult, this was not the case, the ash then consisting not entirely of arsenate, but partly of arsenite. This method gives good results. T. H. P.

Estimation of Arsenic and Phosphorus in Metallurgical Products.

1. Compagno. (*Giorn. Chim. Ind. Appl.*, 1920, 2, 493-497.)—The following simplified modification of the distillation method of Lundin and Blair (Carnot, *Méthodes d'Anal. des Fontes, des Fers et des Aciers*, 1895) is recommended for the estimation of arsenic and phosphorus in steel, etc.: The metal is finely divided, and 5 grms. dissolved in dilute nitric acid (1:1), with the subsequent addition of 10 c.c. of sulphuric acid. The solution is evaporated to dryness, and the residue calcined, ground up, and again calcined until fumes of sulphuric acid appear, and then dissolved in 200 c.c. of hydrochloric acid, and transferred to a distillation apparatus. After reduction of the liquid with 5 grms. of pure copper turnings, the arsenious chloride is distilled into 300 c.c. of water contained in a receiver chilled in ice-water. The arsenic in the distillate is precipitated in the cold by means of hydrogen sulphide, the excess of which is removed by means of carbon dioxide, and the arsenic sulphide collected in a Gooch crucible, washed successively with water, alcohol, and carbon bisulphide, dried and weighed. Phosphorus is estimated by one of the usual methods in an aliquot portion of the residue in the distillation flask, this being first filtered from silica and then oxidised by means of nitric acid and potassium permanganate. If only the phosphorus is to be estimated, the arsenic may be volatilised by treating the hydrochloric acid solution of the metal with hydrobromic acid, or after reduction with pure copper.

Estimation of Titanium and Iron in Rocks. F. Ferrari. (*Giorn. Chim. Ind. Appl.*, 1920, 2, 497-498.)—Titanium and iron are estimated in rocks by separating the silica in the usual manner, reducing the iron in a definite portion of the filtrate from the ferric into the ferrous condition by means of zinc, while a current of carbon dioxide is passed through the flask, and then titrating the titanium with methylene blue. The iron, which is in the ferric condition, is then estimated in another portion of the filtrate by titration with titanous chloride, potassium thiocyanate being used as indicator.

Detection and Estimation of Chromium in Steel. A. Ferni and P. Malaguti. (*Giorn. Chim. Ind. Appl.*, 1920, 2, 559-561.)—The method of detecting and estimating chromium by oxidising it to chromic acid by means of nitric acid and lead peroxide (*Gazz. Chim. Ital.*, 1919, 1, 251) has been adapted to its estimation in steel. One gm. of the sample is dissolved by boiling it with 30 c.c. of dilute (1:4)

nitric acid in a covered beaker, and after nitrous fumes have ceased to be evolved, the solution is treated with 20 c.c. of strong nitric acid (sp. gr. 1.4) and about 1 grm. of lead peroxide, and concentrated to about 4 to 5 c.c. This residue is diluted with 30×35 c.c. of cold water, and then heated nearly to boiling with 25 c.c. of 30 per cent. sodium hydroxide, cooled, diluted to 500 c.c., and filtered. An aliquot part (200 c.c.) of the filtrate is treated with 25 c.c. of 30 per cent. nitric acid (sp. gr. 1.2) which has been freed by prolonged boiling from nitrous acid, and then with 5 to 10 c.c. of 10 per cent. potassium iodide solution, and the liberated iodine is titrated with thiosulphate solution against which has been standardised $\frac{N}{10}$ potassium bichromate solution in the presence of 25 c.c. of 30 per cent. nitric acid.

Composition of Potassium Platinochloride. A. Vürtheim. (*Chem. Weekblad*, 1920, 17, 637-640.)—The double chloride of potassium and platinum retains a trace of moisture even after prolonged heating at 150° C. By precipitation of the salt from its aqueous solution with 96 per cent. alcohol as a very fine crystalline mass it may be obtained almost entirely free from water. The composition of the salt as found by analysis does not quite agree with the formula K_2PtCl_6 , the platinum being slightly higher, and the chlorine lower, than the theoretical amount. In the estimation of potassium as potassium platinochloride an empirical factor must be employed; the value found by the author was 0.1929-0.1930.

W. J. W.

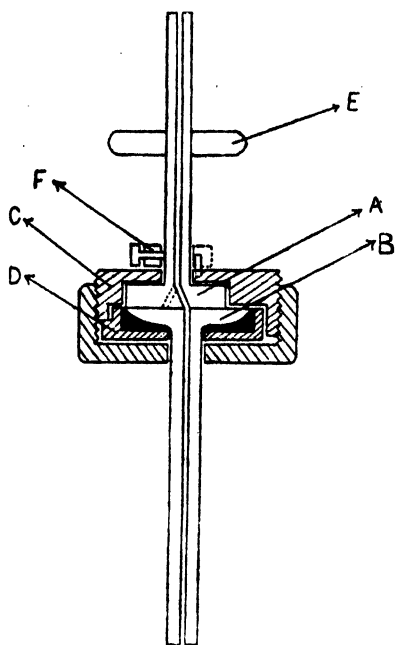
Perchloric Acid as a Dehydrating Agent in the Estimation of Silica. H. H. Willard and W. E. Cake. (*J. Amer. Chem. Soc.*, 1920, 42, 2208-2212.)—The silica in metals and silicates can be rendered insoluble by evaporating the acid solution of the sample with the addition of perchloric acid until dense fumes of the latter acid are evolved. Less silica remains in solution than is the case when the sample is evaporated to dryness with hydrochloric acid.

W. P. S.

Loss of Phosphoric Acid on Heating with Sulphuric Acid or Fusion with Pyrosulphate. W. F. Hillebrand and G. E. F. Lundell. (*J. Amer. Chem. Soc.*, 1920, 42, 2609-2615.)—Losses of phosphoric acid in estimations are due to faulty manipulation, conversion, or volatilisation. Those due to conversion of orthophosphoric acid by heating into the pyro- or metaphosphoric acids, which are incapable of estimation by any methods used for the former, may be rectified by reconversion, by boiling the solution for at least two hours with 10 per cent. sulphuric acid before precipitation. Losses by volatilisation are caused by the employment of excessive quantities of sulphuric acid or sodium pyrosulphate, prolonged heating to secure complete expulsion of sulphuric acid, and evaporation at temperatures above 150° C. In all cases evaporation should cease when fumes begin to appear. No losses occur in the case of evaporation involving only a few drops of sulphuric acid, as in the removal of silica by hydrofluoric acid. Fusion with sodium pyrosulphate should not extend over two hours, nor should the temperature be allowed to exceed that of dull redness.

T. J. W.

APPARATUS, ETC.



Metal-Glass Tap. W. E. Garner. (*J. Soc. Chem. Ind.*, 1920, **39**, 347T-348T.)—A metal-glass tap, for use with a mercury manometer for measuring pressures between 2 and 10 atmospheres, comprises a metal disc A attached to a steel capillary tube and a larger glass disc B fused to a glass capillary, the metal disc being perforated obliquely and the glass disc centrally, the perforations being continuous with the capillaries. The discs are placed in brass cells C and D, keyed together, the glass disc being fixed, but the metal one capable of revolution about an axis parallel to, but not in the same line as, the axis of the glass disc; a strip of metal E soldered to the steel capillary enables the metal disc to be turned. The surfaces of both discs are carefully ground, and a air-tight joint is made with grease. Communication between the discs is obtained in one position only, and the discs may be secured both in the open and closed positions by means of a collar and pin F. W. J. W.

Sulphuric Acid Concentrator and Vacuum Pump. O. Maass. (*J. Amer. Chem. Soc.*, 1920, **42**, 2571-2574.)—The apparatus described consists of an automatic vacuum pump working on the principle of the well-known Töpler pump, but employing sulphuric acid as the motive liquid, this acting at the same time as a powerful absorbent of water vapour. In order to overcome the effect of heat due to the absorption of water, portions of the apparatus containing sulphuric acid are water-jacketed. The apparatus is partially exhausted by means of an auxiliary Geissler pump before use, and the movement of the sulphuric acid is regulated by a control previously described (*J. Amer. Chem. Soc.*, 1919, **41**, 53). The pump was designed to remove water, partially or completely, from large volumes of aqueous solutions, and has been found highly efficient in cases where corrosive and other gases are liberated in addition to water vapour. For details and diagram of the apparatus the original paper should be consulted. T. J. W.

Laboratory Apparatus for Measuring the Permeability and Heat-insulating Capacity of Textiles. G. Colombo. (*Giorn. Chim. Ind. Appl.*, 1920, **2**, 167-168.)—To determine the permeability of textile fabrics to water, the material is stretched across a framework of metal wire, which is fitted into a receiver so as to form a porous bottom, the edges of which are made watertight by means of mercury seals. A volume of water (*e.g.*, 40 mm.) is maintained at a constant level above the stretched fabric, and the quantity which passes through the material in a given

time at a definite temperature is calculated into the volume per sq. cm. Or, in comparing the behaviour of two fabrics, the time in minutes required for the filtration of 500 c.c. of water per sq. cm. under a constant pressure of 40 mm. may be determined. The apparatus for measuring the heat-insulating capacity consists of a cylinder filled with mineral oil and connected with an outside source of electric heat. This cylinder is suspended, by means of the thermometer passing through the cork, in an outer cylinder with perforated walls, so that there is an air space of 15 mm. between the cylinders. The material is stretched round the outer cylinder, the oil is heated to a given temperature, the apparatus placed under a bell jar, and the time required for the temperature to fall through a definite range is recorded.

Apparatus for Determining the Fluidity of Oils at Low Temperatures.

(*Giorn. Chim. Ind. Appl.*, 1920, 2, 388-390.)—The apparatus consists essentially of a U-tube with long arms, 6 m.m. in diameter, upon one of which is a scale graduated in mm. The oil is introduced into this tube up to the zero mark of the scale, and the tube is immersed in a bath, chilled to the desired temperature. A pressure of 50 mm. of water, regulated by a water manometer, is then applied to the other limb of the U-tube, and the degree to which the oil is displaced by the pressure is read upon the scale, and thus affords a measure of the fluidity. For the simultaneous examination of a number of oils, a series of such tubes is placed in a special chamber which contains an observation window, and is connected by means of a valved tube with a cylinder of liquid carbon dioxide, the expansion of which produces the low temperature.



REVIEWS.

PLANTATION RUBBER AND THE TESTING OF RUBBER. By G. STAFFORD WHITBY. Pp. xvi + 559, with 8 Plates and 48 Diagrams. London: Longmans, Green, and Co., 1920. Price 28s. net.

The first part of the work under review deals substantially with the preparation of plantation rubber in its widest aspect, with the character of the various grades produced, and with the chemical and physical nature of the non-rubber constituents (*i.e.*, the resin, and protein, and other serum constituents). Dr. Whitby has collated and handled the subject-matter exhaustively and in scholarly fashion, with the result that Part I. (consisting of 201 pages) may fairly be termed an up-to-date handbook of the Plantation Rubber Industry, with the qualification that the author does not treat of diseases or parasites or of matters other than those which may be claimed as coming within the purview of the rubber chemist and technologist. Part II.—*The Testing of Rubber*—deals essentially with the mechanical and physical character of crude rubber, of the vulcanisation process, and of the vulcanised product. Except and in so far as the determination of "combined sulphur" and the relation of the latter to mechanical and physical properties are concerned, chemistry plays a comparatively

small rôle in the author's scheme; but as he is dealing mainly with the fundamental principles relating to the characteristics of rubber and its behaviour towards vulcanisation with sulphur only, and not with rubber manufacture proper, no other course could well have been expected. As Dr. Whitby correctly points out, "Vulcanised rubber is in most connections technically valuable because of its mechanical properties. Hence, speaking broadly, the mechanical behaviour of the rubber after vulcanisation must be the final criterion for the purpose of evaluation. Chemical analysis, not only in the case of raw rubber, but also in the case of vulcanised rubber, will, finally, be of value only in so far as it can be correlated with the mechanical behaviour of the vulcanised rubber." The author has rendered a great service to those interested in rubber science and technology by collating and criticising the numerous researches on physical and mechanical characteristics and on vulcanisation published over the past century, researches frequently difficult of access and hitherto sadly in want of critical review. The merit of the work in this connection is enhanced by a most excellently compiled Bibliography. In conclusion, it should be stated that Dr. Whitby's book is one of the series of "Monographs on Industrial Chemistry," edited by Sir Edward Thorpe, C.B., F.R.S., and that the printing and illustrations are first-rate.

PHILIP SCHIDROWITZ.

THE DETERMINATION OF HYDROGEN IONS. By W. MANSFIELD CLARK, M.A., Ph.D.
Baltimore, U.S.A.: Williams and Wilkins Company, 1920. Price \$5.50.

A glance through the contents of this book shows that Dr. Clark could have chosen no portrait to face the frontispiece of his book more appropriate than that of S. P. L. Sørensen, the Director of the Carlsberg Laboratorium at Copenhagen. The lucid exposition of the relationships governing the dissociation of water in which electrolytes are dissolved, contained in the opening chapters, is developed *ab initio* from Sørensen's treatise published in the Carlsberg Laboratory Reports. For those who have not had the good fortune to work with the new Clark and Lubs series of indicators, it is instructive to note their nuances by the show of tints portrayed in excellent reproduction in colour. These pages are more ambitious than any previously published on this subject.

Passing through the book from chapter to chapter the pleasure increases in the thoroughness and practical insight of the author into this fascinating field of study. No phase is treated laboriously, yet all the essential matter is there and in detail—standard buffer solutions for colorimetric comparison (Chapter V.); the protein error and the salt error in colorimetric determinations; potential differences at liquid junctions (a difficult chapter this to write, but here excellently treated); the relation of hydrogen electrode potentials to reduction potentials; sources of error in electro measurement of P_H , etc., etc. These are all excellent, and not only the general theory and the details of its application to every-day laboratory practice, but the numerous details of equipment are dealt with in a manner that compels admiration and the desire that this book should be widely circulated to our more complete knowledge and advancement.

It is difficult to find any point of criticism. Chapter XIX., dealing with applica-

tions of the methods to the study of biochemical problems, is historical, condensed, to the point, and suggestive, and not less valuable is a bibliography of some 1,200 entries which for reference is of inestimable value.

G. S. WALPOLE.

MARGARINE. By WILLIAM CLAYTON, M.Sc., A.I.C. Monographs on Industrial Chemistry. Pp. 187. London: Longmans, Green and Co., 1920. Price 14s.

This monograph being undoubtedly, as the author states in the Preface, the first of its kind to be published in any language, it is natural to approach it with some considerable interest on account of the extraordinary secrecy and mysteriousness which have surrounded the manufacture of margarine, a manufacture which in itself is very simple. Its claim too, of course, only holds good in so far that the subject-matter is confined to margarine, as the section on Margarine in Hefter's "Technologie der Fette und Oele" contains a remarkably good history of the manufacture, etc., of margarine up to the date of its publication, and similar, if more condensed, articles are to be found elsewhere. It is, however, quite true that this present work is the only one extant which brings the history of margarine up to the present day, and for this reason must command considerable attention on account of the well-known experience of the writer.

The author has certainly succeeded in giving a concise account of the processes which are used in the best equipped margarine factories, though many undoubtedly will feel sorry that he has kept so carefully to the rules laid down in the Introduction—viz., that these monographs are not to be concerned with the technical minutiae of manufacture—as a rather more detailed account might have been hoped for.

In the chapter dealing with the oils and fats used in margarine manufacture, attention is drawn to nearly all the oils which have from time to time been used. The descriptions, etc., are clear, lucid, and correct, though, at the moment, the manufacture of margarine is confined to a comparatively few varieties of oils and fats.

It is doubtful if many manufacturers would agree that soya oil is admirably suited for margarine, and it is improbable that shea nut oil has found its way into the manufacture, except in very small quantities, as the refining of this oil is a somewhat difficult process. Some useful information, however, is given with regard to the use of hydrogenated oils, though the author appears to be somewhat over hopeful about the use of these fats in margarine manufacture, as, for many reasons, they have not proved as desirable as was at first hoped.

A chapter follows on the examination of the milk used in margarine manufacture. This is not as reliable in regard to detail as it might be, and, though useful to a trained chemist, might lead to unfortunate conclusions in the case of the less experienced works chemist. For instance, the test given for the presence of formaldehyde sometimes gives a coloration with pure milk. Also the use of vaseline in the test for *Bacillus enteritidis sporogenes* is to avoid anaerobic conditions of culture which are stated to be necessary. Again, among the tests for pasteurised milk, the cream-lime test is of doubtful utility, nor is it by any means possible to determine the method of pasteurisation by incubation of samples of the treated milk. It is necessary to draw attention also to the extraordinary differences in temperature

which are spoken of as being used in the preparation of milk for culture purposes. For instance, the temperature of "flash" pasteurisation is given in one place as 82° F., in another as 71° F., and for bulk pasteurisation as 70° C. for thirty to forty minutes, and in another place 60° to 65° C. for thirty minutes. The methods of pasteurisation are well dealt with, but few people will agree with the author that the "Tube" pasteuriser described on page 42 is the most satisfactory for "Holder" pasteurisation. When, however, one turns to the practical preparation of starters, one notes immediately that the temperatures given as being employed in sterilisation or pasteurising milk for such cultures are those of the practical man, and are very much more satisfactory in practice than those given in the theoretical discussion of the subject.

The section dealing with the inoculation and ripening of the milk for culture work is undoubtedly good, though, of course, ideas on this subject vary with different workers. The sections dealing with the actual manufacture of margarine are quite up to date, and well describe the modern methods of emulsifying, working, etc., though the statement that the older method of cooling by ice-spray washes out some of the milk is not true when emulsification has been correctly carried out. A chapter on the theory of emulsification will be of great interest to those engaged in the scientific control of margarine manufacture, and is distinctly helpful and suggestive of new lines of work.

The sections on the analysis of butter and margarine give a concise and adequate account of the methods usually employed for the analysis of margarine, and show careful discrimination. The section on deterioration in storage would perhaps have been somewhat more useful if it had dealt with the deterioration of margarine rather than that of butter, but, as little work has been published on this matter, it would be too much to expect any exhaustive treatment of the subject, though many will disagree with the author with regard to the inefficiency of boron compounds as a preservative, when properly applied. A useful epitome of work on the question of vitamins is added, and the work concludes with an exceedingly useful and full bibliography of papers, etc., published in connection with margarine and butter, the value of which cannot be over-estimated, and on the compilation of which the author is to be greatly congratulated.

As with all the other monographs of this series, the work is well printed, and this is exceedingly free from any serious errors.

CECIL REVIS.

BIOCHEMICAL CATALYSTS IN LIFE AND INDUSTRY: PROTEOLYTIC ENZYMES. By JEAN EFFRONT. London: Chapman and Hall, Ltd. 1917. Price 33s.

The sub-title of this treatise indicates its contents more clearly than the main title, for indeed it is only the proteolytic enzymes that are dealt with. The main subject-matter is divided into the coagulating enzymes (thrombin, myosinase, rennet); pepsin; trypsin; and erepsins, these being treated more or less from the standpoint of the physiologist and biological chemist. Although the treatment is modern, the translator has unfortunately not been able to bring the subject-matter up to date. For instance, in dealing with the coagulating enzymes, the work of Bordet, Morawitz,

and the earlier work of Mellanby, are collated and carefully discussed, but the later work of Dale (1916), which puts the whole theory of blood coagulation on a more secure footing, is not mentioned. The chapters dealing with trypsin and pepsin give the impression of systematic reading and collation, but of no particular intimacy with the subject from an experimental point of view. Similarly, that portion of the book (roughly about one-quarter) dealing with the erepsins is not particularly inspiring, although the subject is treated at greater length than is usually the case.

There are bibliographies at the end of each chapter, but the index is fragmentary and of little value.

G. S. WALPOLE.

THE ANALYST'S LABORATORY COMPANION. By ALFRED E. JOHNSON, B.Sc., F.I.C.
Pp. 176. London: J. and A. Churchill. 1920. Price 10s. 6d. net.

Practising chemists will welcome a new edition of this little handbook for the laboratory, for it gives in a form convenient for reference physical and chemical data constantly required in analytical work.

The tabular matter, details of the preparation of reagents, reports of Commissions on chemical subjects, statutory regulations, etc., have, in the main, been brought up to date, but there are still numerous important omissions. For instance, the strength and purity of glacial acetic acid can be most accurately estimated by the determination of the freezing-point, but Riidorff's table is not given; nor is there even an abridged version of Oudemann's table, showing the relationship between the strength and specific gravity of acetic acid.

The notes on specialised branches of applied chemistry are less satisfactory, at all events in some cases, than the general matter. A chemist who has to deal with a problem outside his ordinary routine work would not consult this book for his information, but would go direct to a treatise on the subject. On the other hand, the specialist ought to be able to refer to this handbook as a *memoria technica*. If, for instance, we apply this criterion to the section on Oils and Fats, it will be found that some of the information is superfluous, whilst other data are lacking. For example, an oil chemist does not require a definition of the iodine value, but he does need to refresh his memory as to the proportion of the ingredients in Wijs' solution, or of the quantities of reagents to be used in Twitchell's rosin test, and such details as these are not given. Again, the analytical values quoted are not sufficiently extensive, and should have included all the oils and fats of commercial importance, whereas even an oil of such frequent occurrence as menhaden oil is omitted. Dalican's method or the "titre" test, for which the author, in common with many other chemists, adopts the ugly American translation "titer" test, is not sufficiently described to be of use to the chemist.

A similar criticism may be passed upon other sections, and it would greatly have increased the value of the book if these had been revised by specialists in each subject. It is with the object of improving the next edition that these suggestive criticisms are made.

EDITOR.

THE CENTENARY VOLUME OF CHARLES GRIFFIN AND COMPANY LTD., PUBLISHERS,
1820-1920.

Every chemist has upon his shelves one or more volumes published by Messrs. Charles Griffin and Co., and will therefore feel interested in a firm the history of which has run parallel with the development of chemistry as a practical science.

When the firm began its career in 1820 in Glasgow, it gave its chief attention to works of general literature, but it was not long before it began to specialise in technical and scientific subjects, its first chemical venture being *Chemical Recreations* by C. J. Griffin, which was published in 1826. This was followed in 1832 by Rose's *Manual of Analytical Chemistry*, and from that time onwards there has been a steady succession of technical works until, at the present time, there is practically no branch of applied science in connection with which thoroughly practical books have not been published by this house.

With regard to chemical literature it is interesting to note that Messrs. Charles Griffin and Co. were originally responsible for the publication of the first weekly journal devoted to chemistry—*The Chemical News*.

The book which has been issued to commemorate the centenary of the firm is an admirable specimen of the printer's and bookbinder's craft, and one lays it down with regret that it is not possible to have all one's books produced with the same distinction. The book opens with a general Introduction by Lord Moulton, who points out how indispensable to progress in any science is an efficient supply of trustworthy textbooks, and lays stress upon the services that the firm was able to render the country during the war, owing to its being in an exceptional position to supply such books.

After a brief account of the beginning of the business, the progress marked in the different branches of science by the publication of successive books is described in readable outline by authorities on their respective subjects. The metallurgical section is dealt with by Professor Gowland; mining, the production of petroleum, and geology by Professor Louis; and chemical technology by Dr. H. Hodgson, whose critical description of the development of the dyestuff industry is particularly interesting. The other sections on such subjects as textiles, and medicine and allied sciences, are also far from being mere catalogues of books.

Chemists will congratulate Messrs. Charles Griffin and Co. upon the celebration of their centenary, and acknowledge the assistance they have received from their publications, many of which they owe to the foresight of Mr. Francis J. Blight, who for over twenty-five years has controlled the destinies of the firm.

ORGANIC CHEMISTRY FOR MEDICAL, INTERMEDIATE SCIENCE AND PHARMACEUTICAL STUDENTS. By A. KILLEN MACBETH, M.A., D.Sc., F.I.C., etc. Longmans, Green and Co. Price, 6s. 6d. net.

This little book of 226 pages is interesting in view of the very large number of substances dealt with. When it is considered that the chief families of open chain bodies and cyclic substances, the sugars, glucosides, terpenes, alkaloids, stereo-isomerism, geometrical isomerism, oxime isomerism, proteins, etc., etc., are studied,

one begins to wonder how it can be done in such a very small compass. Our author's method is to concentrate and evaporate almost to dryness. In twenty-four pages he summarises on fats, carbohydrates, proteins, alkaloids, and terpenes. It is more of a notebook than a textbook, and to appreciate it properly a student should have a fair knowledge of organic chemistry. There are points that will require correcting in a future edition. On p. 98 CH_3NNOH should be placed in brackets to denote a probability, otherwise it is misleading. In dealing with acetoacetic ester, p. 108, some reference should be made to the two forms of this body, as in one equation it is written in the keto form, and in a more detailed view of its formation the enolic form is used. This is apt to be very confusing to a beginner.

There is a rather serious error where the formula for fructosazone is given on p. 159. It should be the same as glucosazone on the opposite page, and not as represented. On p. 192 it is stated that aniline treated with nitrous acid gives the diazonium hydrate. As a matter of fact, diazoamidobenzene is obtained. This error is repeated on p. 197. On p. 194, dealing with the detection of a primary aromatic amine the aniline should be dissolved in dilute HCl and well cooled before adding sodium nitrite and coupling with the phenol.

In the formation of diphenylamine from aniline and iodobenzene, on p. 195, the high temperature necessary for this reaction, somewhere about 400°C ., should have been mentioned. Again, in the preparation of methyl orange, p. 202, allusion might with advantage have been made to the use of sodium carbonate for dissolving the sulphanilic acid.

The book is very sketchy in character, but is probably sufficient for medical and pharmaceutical students who are supposed to have a profound knowledge of important and complicated organic compounds after about six months' study. Yet notwithstanding its limitations, it is very well arranged, concise, and to the point, and can be confidently recommended. Another good feature of the book is the schemes showing the relationship between the various organic substances. For example, starting with acetic acid, it is shown schematically how malonic, succinic, glycollic acids, etc., may be derived from it.

This is excellent, and it is only by constant practice in this way, backwards and forwards from one family of substances to another, that the student realises the connection between the various compounds, remembers general reactions, and obtains a true insight into the fascinating study of the chemistry of carbon compounds.

F. H. STREATFEILD.



THE INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

PASS LIST.

JANUARY (1921) EXAMINATIONS.

At the Examinations of the Institute of Chemistry held recently, Mr. Keith Alister Johnstone McClure, B.A. (Cantab.), passed in General Chemistry, and Mr. Cecil Chapman passed in Chemical Technology, with special reference to Coal Tar and Ammonia.

Both Candidates have been elected to the Associateship of the Institute.



APPOINTMENT OF PUBLIC ANALYST FOR THE COUNTY BOROUGH OF BRIGHTON.

The following correspondence and report have been received for publication from the Public Appointments Committee of the Institute of Chemistry :

THE TOWN CLERK,
TOWN HALL, BRIGHTON.

January 26, 1921.

SIR,

The attention of the Councils of the Institute of Chemistry and of the Society of Public Analysts has been directed to the conditions laid down by your authority for the appointment of Public Analyst for the County Borough of Brighton.

The Councils of the Institute and of the Society desire me to direct attention to the fact that the remuneration offered is far below that now generally obtained by Public Analysts in Great Britain. Assuming a reasonable proportion of milks and allowing for the salary of £50, it would appear that the average fee per sample is not more than 8s. 6d., while the average fee paid by all authorities throughout the country is approximately 15s.

The Councils would venture to remind your authority that apart from the advanced cost of living, the cost of assistance, apparatus, and materials has more than doubled. All of this must be borne by the Analyst, and would render it practically impossible, on the remuneration proposed, for him to carry out the duties of the office satisfactorily and with any margin of profit to himself. The Councils are aware, moreover, that several Public Analysts whose qualifications have already been approved by the Ministry of Health have declined to become candidates for the appointment. The number of candidates likely to apply for the appointment is therefore limited in a manner which is not in the best interests of the proper administration of the Sale of Food and Drugs Acts.

I am requested to ask you kindly to bring this letter to the notice of your Committee, and to express, on behalf of the Councils of the Institute and of the Society, the hope that your authority will consider the revision of the conditions before proceeding further with the appointment.

Yours faithfully,

(Signed) G. S. W. MARLOW
(Assistant Secretary, the Institute of Chemistry).

REMUNERATION OF PUBLIC ANALYSTS.

The Councils of the Institute of Chemistry and of the Society of Public Analysts have invited Public Analysts to provide data as to the remuneration and other conditions of their appointments in 1914 and in 1920 respectively.

Replies were received in respect of 140 appointments in England and Wales, and in respect of 102 appointments in Scotland; of these, 9 and 1 respectively were whole-time officials.

PART-TIME OFFICIALS.

Public Analysts receive remuneration—

(Class A) by payment of an agreed fee per sample.

(Class B) by payment of an agreed salary, which is intended to cover the analysis of all samples of foods and drugs.

(Class C) by payment of a retaining fee, together with an agreed fee for each sample of foods and drugs.

(Class D) by payment of a retaining fee or salary in consideration of the analysis of an agreed number of samples, and by fee for the analysis of all samples in excess of that number.

The number of appointments held under each of the above classifications was :

	England and Wales.	Scotland.	United Kingdom.
Class A	56	26	82
Class B	7	2	9
Class C	63	59	122
Class D	5	14	19
	131	101	232

For the purposes of comparison, the total actual remuneration (including the proper proportion of the salary or retaining fee) for the analysis of each sample was calculated. The figures for the average remuneration are included in Table A (p. 78), which shows the remuneration received in 1914 and 1920 respectively. The table shows also the average remuneration per sample which applies to those appointments of which the conditions have remained unaltered since 1914, and the average remuneration of those in which the fee, retaining fee, or salary has been increased.

In a few cases in which the retaining fee or salary had not been altered since 1914, a reduction in the number of samples caused the remuneration per sample to increase, whilst the payment received by the Analyst had remained stationary or had decreased. Those appointments were treated in the tables as if their terms had remained unaltered.

Table B shows similar figures for the more important appointments (from which at least 100 samples were received during 1920).

Owing to the disturbed state of Ireland, no useful data can be given for that country.

WHOLE-TIME OFFICIALS.

Replies were received from 10 whole-time salaried Analysts. Of these 5 were required to perform analyses of samples other than foods and drugs, together with bacteriological examinations. The salaries of the other officials varied according to

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the number of samples submitted, but there was no possibility of assessing an average salary according to the number of samples.

The following table will, however, serve as a rough guide :

	Average Salary—		Average Increase per Cent.
	In 1914.	In 1920.	
	£	£	
Appointments with less than 100 samples (of all kinds) per annum	287	658	122
Appointments with more than 1,500 samples (of all kinds) per annum	620	1,036	67

TABLE A.—ALL APPOINTMENTS.

Total remuneration per sample of foods and drugs submitted to Public Analysts. (Average from 232 appointments: 181 in England and Wales, 101 in Scotland.)

	United Kingdom.	England and Wales.	Scotland.
	Average.	Average.	Average.
Fees in 1914	10s. 6d.	11s. 0d.	9s. 10d.
Fees in 1920	15s. 8d.	14s. 6d.	17s. 4d.
Fees which have not been increased since 1914	12s. 10d.	12s. 11d.	12s. 9d.
Fees which have been increased since 1914 (1920)	16s. 2d.	14s. 11d.	17s. 8d.

The fees have not been increased in the cases of 34 appointments in England and Wales and 12 appointments in Scotland.

TABLE B.—APPOINTMENTS FROM WHICH THE SAMPLES IN 1920 EXCEEDED 100.

Total remuneration per sample of foods and drugs submitted to Public Analysts. (Average from 98 appointments: 86 in England and Wales, 12 in Scotland.)

	United Kingdom.	England and Wales.	Scotland.
	Average.	Average.	Average.
Fees in 1914	10s. 7d.	10s. 8d.	10s. 3d.
Fees in 1920	14s. 8d.	14s. 3d.	17s. 2d.
Fees which have not been increased since 1914	12s. 1d.	12s. 1d.	—
Fees which have been increased since 1914 (1920)	15s. 1d.	14s. 9d.	17s. 2d.

The fees have not been increased in the cases of 17 appointments in England and Wales. They have all been increased in Scotland.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

ANNUAL GENERAL MEETING AND ORDINARY MEETING, HELD
FEBRUARY 2, 1921.

THE Annual General Meeting of the Society was held at the Chemical Society's Rooms, Burlington House, W. 1. The President, Mr. Alfred Smetham, occupied the chair.

The minutes of the previous Annual General Meeting were read and confirmed.

Miss D. Hewer and Captain E. J. Lush were appointed scrutators of the ballot papers for the election of officers and Council for 1921.

The Hon. Treasurer presented the accounts of the Society for 1920, and votes of thanks were passed to the Hon. Treasurer, the Acting Hon. Treasurer (Mr. E. W. Voelcker), and the Hon. Secretaries.

Messrs. Marreco, Houseman, and Brandon, chartered accountants, were appointed Auditors of the Society's accounts for 1921.

The President delivered his Annual Address. Mr. G. R. Thompson proposed that a hearty vote of thanks be accorded to the President for his address and for his services in the chair, and that his permission be asked to print the address in the ANALYST. This was seconded by Dr. Bernard Dyer, and the motion was carried.

The scrutators having reported the result of their examination of the ballot papers, the President announced that the officers and Council for 1921 had been elected in accordance with the Council's nominations as follows:

President.—Alfred Smetham.

Past Presidents serving on the Council (limited by the Society's Articles of Association to eight in number).—Leonard Archbutt, Edward J. Bevan, A. Chaston Chapman, Bernard Dyer, Otto Hehner, Samuel Rideal, E. W. Voelcker, J. Augustus Voelcker.

Vice-Presidents.—W. J. A. Butterfield, C. A. Keane, G. Rudd Thompson.

Hon. Treasurer.—Edward Hinks.

Hon. Secretaries.—P. A. Ellis Richards, E. Richards Bolton.

Other Members of the Council.—F. W. F. Arnaud, B. A. Burrell, F. H. Carr, R. L. Collett, C. H. Cribb, Norman Evers, P. J. Fryer, J. H. B. Jenkins, S. E. Melling, G. W. Monier-Williams, Raymond Ross, C. J. H. Stock.

An Ordinary Meeting followed the Annual Meeting, the President, Mr. Alfred Smetham, F.I.C., being in the chair.

The minutes of the previous Ordinary Meeting were read and confirmed.

Certificates were read for the first time in favour of Messrs. Jules Cofman-Nicoresti, Walter K. Fletcher, William Singleton, Francis G. H. Tate, James Darnell Granger, Ph.D., F.I.C., Russell G. Pelly, F.I.C., and Edward B. Maxted, Ph.D. (Berlin), B.Sc. (Lond.).

A Certificate was read for the second time in favour of Mr. W. R. Schoeller, Ph.D.

The following were elected Members of the Society: Messrs. Urban Aspey, Herbert Corner Reynard, B.Sc. (Lond.), A.I.C., Edwin Burnhope Hughes, B.Sc. (Lond.), A.I.C., Harry Jephcott, M.Sc. (Lond.), A.I.C., and Arnold Lees, A.I.C.

The following papers were read: "Extract of Red Squill (*Scilla maritima*) as a Rat Poison," by F. W. Smith, B.Sc., A.I.C.; "Iodimetric Determination of the Diastatic Power of Malts," by Julian L. Baker, F.I.C., and H. F. E. Hulton, F.I.C.; and "The Composition of Harrogate Mineral Waters," by W. Lowson, B.Sc., F.I.C.



OBITUARY.

It is with great regret that we have to record the following deaths:

Professor Émile Bourquelot (January 28).

Dr. J. C. Cain, Editor of the *Journal of the Chemical Society* (January 31).

Sir Charles A. Cameron, Past-President of the Society of Public Analysts (February 27).

Dr. William Odling, Hon. Member of the Society (February 17).

Obituary notices will be published in a forthcoming issue.



THE MINISTRY OF FOOD.

THE Prime Minister announced in the House of Commons on February 17 that the Ministry of Food would be brought to an end before March 31.

The Food Orders still remaining in force include the following: Dripping (standard of quality), 1919; Edible Fats (standard of quality), 1919; Manufacture of Flour and Bread, 1918.

ANNUAL ADDRESS OF THE PRESIDENT.

(Delivered at the Annual General Meeting, February 2, 1921.)

It has been customary in the past for the President of the Society at the Annual General Meeting to bring under survey the activities of the members and the work and progress of the Society in the official year that sees its close at the Annual Meeting; and it thus falls to my lot to-day to review the proceedings of the past year. Following as I do in the footsteps of a long array of brilliant chemists and able administrators, I feel that the task of doing adequate justice to the subject is no light one, and I would that I could relegate the duty now imposed upon me to someone better fitted for the post; but since the address must in the main be a résumé of the work of others rather than an exposition of one's own views, I have greater confidence than I should otherwise feel in imposing my remarks upon you.

During the year ending December 31, 1920, the total number of members, including eleven honorary members, has increased from 448 to 454. New members elected number twenty-six, while we have lost eight members by death, eight members have resigned for various reasons, while three members have been removed for non-payment of their subscriptions.

The names of the deceased members whom we mourn are: William Beaverly Cowie, Walter William Fisher, James Wright Gatehouse, Alfred Howard, John Ruffle, Robert Tervet, Elwyn Waller (of New Jersey, U.S.A.), and Samuel Archibald Vasey.

Obituary notices have appeared in the pages of the ANALYST of Walter William Fisher and James Wright Gatehouse.

Walter William Fisher was one of the oldest members of the Society of Public Analysts, and acted as its President in 1889 and 1890. He was Public Analyst for the counties of Oxford, Berks, and Bucks, as well as for the City of Oxford, which posts he held for nearly forty years, and to within a week of his death carried out actively the duties appertaining to the posts. We deplore his death, and the many members of the Society who knew him have to lament the loss of a staunch friend.

James Wright Gatehouse died on October 16, 1920, at Bath, at the age of seventy-nine. In 1877 he was appointed Public Analyst for the City of Bath, and, eleven years later, Public Analyst for Wiltshire. In addition to his duties as Public Analyst, he carried on a private practice and did much good work in various investigations.

By the death of John Ruffle I have lost a friend of over forty years standing. Although of a gentle and retiring nature he was a man of many parts, and did much useful work. He will be best remembered, perhaps, as the originator of the Ruffle method for the estimation of nitrates by the combustion process.

During the session twenty-three papers were read, while thirty-two papers and eight "notes" were published in the ANALYST.

- During the year 1920 the following papers appeared in the pages of the ANALYST :
- "A New Method for Detecting Adulteration in Butter and for Estimating Fats of the Coconut Group." By George Van B. Gilmour.
 - "A New Process for the Estimation of Arsenic, with Notes on the Chemistry of the Marsh-Berzelius Process." By B. S. Evans.
 - "The Estimation of Mercury." By H. B. Gordon.
 - "An Investigation into the Composition of the Unsaturated Hydrocarbons present in Coal Gas." By F. S. Sinnatt and L. Slater.
 - "The Estimation of the Available Oxygen in Sodium Perborate and in Perborate Soap Powders." By H. Trickett.
 - "The Differentiation between Poor and Adulterated Milk." By T. R. Hodgson.
 - "Machine for Washing Precipitates." By Eric Sinkinson.
 - "The Detection of Finger-Prints on Documents." By C. Ainsworth Mitchell.
 - "Photomicrography with Simple Apparatus." By Thomas J. Ward.
 - "Note on the Solubilities of Theobromine." By Raymond V. Wadsworth.
 - "A New Method for the Estimation of Methyl Alcohol." By S. B. Schryver and Cyril Christian Wood.
 - "Note on Opium Poisoning." By E. H. Hankin and D. Chatterji.
 - "An Examination of Certain Milk Records." By G. W. Monier Williams.
 - "An Improved Slide Rule for Dairy Calculations." By H. Droop Richmond.
 - "The Examination of Chinese Crude Camphor." By E. R. Dovey.
 - "Estimation of the Age of Ink in Writing." By C. Ainsworth Mitchell.
 - "A Rapid Volumetric Method for the Estimation of Iron, Applicable in the Presence of Hydrochloric Acid, Phosphorus, Oxy-acids, and Organic Matter." By H. Droop Richmond and Edith M. Ison.
 - "The Estimation of Nitroglycerin." By H. Droop Richmond.
 - "The Effect of Pressure upon the Polenske and Reichert-Meissl Values." By Vincent H. Kirkham.
 - "The Composition of Milk in British East Africa." By Vincent H. Kirkham and A. C. Barnes.
 - "Note on the Refractive Indices of Mixtures of Isopropyl Alcohol and Acetone." By Dorothy Muriel Palmer.
 - "The Estimation of Codeine." By H. E. Annett and Haridas Sen.
 - "The Influence of the Free Fatty-Acid Content in the Valuation of Chinese Wood Oil by the Browne Polymerisation Test." By Philip E. Jameson.
 - "Apparatus for Evolution Methods of Analysis." By E. R. Dovey.
 - "An Improved Form of U-tube." By E. R. Dovey.
 - "A Study of the Determination of Potassium as the Perchlorate, and the Separation from Sodium, etc." By R. Leitch Morris.
 - "Notes on Rubber Analysis." By A. R. Pearson.
 - "An Improvement in the Haldane General Air Analysis Apparatus." By Robert C. Frederick.
 - "The Gravimetric Estimation of Bismuth as Phosphate and its Application in Ore Analysis." By W. R. Schoeller and E. F. Waterhouse.
 - "The Position of Analytical Chemistry in France." By L. Cofman.
 - "Statutory Standard for Ghee." By T. K. Ghose.

In addition to the papers published, the "Notes" on analytical methods and other matters have proved exceedingly interesting and useful, and I welcome this comparatively new departure as a distinct boon to analysts generally.

During the year thirty-two books have been reviewed in the pages of the ANALYST. In my opinion these Reviews have been wholly admirable. They have been fearlessly and faithfully done by experts in the subjects reviewed whose judgment is reliable. They have brought to my notice some books of sterling value, and in other cases have saved my pockets from being depleted of cash and my bookshelves from being overloaded with trash.

Before leaving the subject of the publications in the ANALYST, I cannot refrain from mentioning the loss which the Society has sustained by the resignation of Mr. Julian L. Baker as Editor of the ANALYST to assume the Editorship of the *Journal of the Institute of Brewing*. Mr. Baker has been Editor of the ANALYST for thirteen years, during the whole of which time the Journal has been admirably conducted, and each succeeding year, thanks to his assiduous attention and genius, has seen, I think it will generally be admitted, an improvement on the previous ones. Bowing to the inevitable, we very reluctantly relinquish him, knowing that what is our loss is his and others' gain.

Fortunately, we have been able to secure in Mr. Baker's stead a new Editor, Mr. C. A. Mitchell, who is well known to us by his scientific abilities, his literary merits, and his devotion to the Society and its publication. Mr. Mitchell as an author has a high reputation, and he has been an Abstractor for the ANALYST for a long time. To him we may look with confidence to maintain the high standard and traditions of our Journal.

It was with much satisfaction that we welcomed back our Hon. Treasurer, Mr. E. Hinks, who has returned safe and sound to resume his duties. At the same time I ought to voice the thanks of the Society to Mr. E. W. Voelcker as Acting Treasurer during Mr. Hinks's absence.

Before leaving this part of my subject, I should like to take this opportunity of congratulating one of our past Presidents (Mr. A. Chaston Chapman) on his election as a Fellow of the Royal Society.

We have to regret the prospective loss, by absence abroad—we hope for a comparatively short period only—of our old and faithful friend, Otto Hehner. Mr. Hehner is too well known to all of us to render it necessary for me to recall to your minds all the posts he has held and all the work he has done to further the interests of the Society and to advance the cause of analytical chemistry.

Of the many questions which have come before your Council for consideration one has been a motion to ascertain whether the present somewhat cumbrous title could not be modified in such a way as to include all the present members, while at the same time the prestige of nearly fifty years' strenuous work and endeavour under the old title could be preserved. Legal advice has been taken, and I understand that there is no legal difficulty in altering the name of the Society, and at the same time of preserving the privileges which we at present enjoy. It is not an easy matter to select a title which will be at once descriptive, inclusive, and euphonic; but there should be no insuperable difficulty in finding a solution.

The original title of the Society of Public Analysts has become so widely known and respected that in many ways it seems a pity to hide its identity under a new title : but as the term public analyst is limited by Act of Parliament to those analysts appointed under the Food and Drugs Act—and cannot, therefore, be appropriated by practising analysts generally—there seems no alternative but to change the name or continue as at present. Doubtless some happy solution of the problem will be evolved, but of the suggestions hitherto made none seem quite to meet my æsthetic taste. There is a crispness about our original title which seems to arrest the attention at once.

The fact that the proportion of analysts other than public analysts has been steadily increasing renders the alteration in the title, if it is to be descriptive of the objects of the Society, the more necessary, and as the number of public analysts is practically stationary, while the analysts engaged in general practice and in connection with trade and manufactures are steadily increasing and, with returning trade and prosperity, are bound to increase rapidly, it seems to me that if the alteration in title is necessary or desirable the present is an opportune time to make it.

I have said that the proportion of analysts other than public analysts is in the future likely to increase at a rapid rate, and I am basing the statement on a careful observation of commercial and manufacturing conditions for a period of over forty years. When I went into practice my work consisted for the most part of analyses to assist in the control of manufacturing processes, or to help purchasers to make a selection of produce sold practically on a flat rate : and very few of the analyses were sent under contracts, the price to be decided by the contents of the valuable constituent or constituents, as the case might be.

As the years went on and rule of thumb in manufactures was replaced by more scientific control, purchasers insisted on safeguards, especially when buying forward, and by gradual evolution in nearly every branch of industry. Safeguards in contracts are inserted, and in many instances these safeguards take the form of restrictions based upon analytical control.

Concurrently with the altered conditions in manufacturing methods, and the greater demand for more definite specifications with regard to quality, especially when the goods in question have been bought to arrive, the demand has arisen for increasing the safeguards against any inferiority in the produce which may be tendered in fulfilment of a contract. This desire on the part of the more progressive merchants and manufacturers has brought into existence a considerable number of combinations of diverse interests, who, forming themselves into associations representing their respective trades or businesses, have generally controlled the transactions, especially in the case of our overseas trade. Each year sees the framing of innumerable new contract forms, and the modification of old ones, and with the increase in the stringency of the demands on the part of buyers there has been necessarily an increase in the volumes of work falling to the lot of analysts in general. And if I read the signs of the time aright it seems to me that the tide having begun to flow in this direction it will continue to do so until a high-water mark has been reached and practically the whole of our commerce brought under scientific control. The employment of men of exceptional talent by companies and associations for the

pursuit of pure research, in contradistinction to the control of manufacturing processes or business transactions, is evidence sufficient to indicate that our leaders of commerce and industry are rapidly realising, thanks partly to work done by chemists and other scientists during the war, that research work is not only a necessary adjunct to a successful business enterprise, but is at the same time a highly profitable investment.

And I am glad to see this tendency on the part of leading business men to call to their assistance the best brains of our profession, because it must inevitably have a leavening effect on the general mass of business men who, in the past, have regarded chemists, not as a necessary controlling influence in the factory or in business, but as an unavoidable adjunct to the business side of their concerns.

The admirable work done during the war by so many members of our profession, both at home and abroad, and the important part played by the investigations and work of our chemists in finally winning the war, has brought home to the nation at large the immense importance and possibilities of chemistry, not only in fostering the interests of our own industries, but in advancing the cause of civilisation the world over. That the ever-increasing developments of chemistry should have been so directed as to have caused such world-wide havoc is greatly to be deplored, and it is to be hoped that the effects produced and the lessons learned will have throughout the ages the effect of teaching future generations the folly of diverting our beloved science from the arts of peace to those of war.

But to revert to my theme. The formation of the numerous bodies and associations, which I have referred to, has increased the volume of analytical work largely, and to that extent it is a good thing for the profession generally, but as the effect, so far as the commercial side of our work is concerned, is to increase the number of analyses of a similar nature there is a tendency to render a good deal of our work tedious and to some extent monotonous. In the nature of things such a result is almost inevitable, as with greater proficiency and greater experience in any particular direction the confidence of one's clients in one's work is sure to grow and, snowball-like, the volume of work is sure to increase, and thus the routine of the laboratory is too apt to assume the functions of something approaching a factory rather than the purely investigational work which we usually associate with a scientific laboratory.

In this respect, however, we are not far removed from the specialists in medicine or surgery who, after a long preliminary training, find themselves in the hey-day of their prosperity dealing with special cases day in and day out.

There is, of course, in addition to the purely routine side of a practice, a considerable amount of work of a highly technical and exceedingly interesting nature which breaks the monotony of life and gives a zest to one's daily occupations.

The point which I am aiming to make clear is that, although the tendency of the age is to make specialists of us all, care must be taken to found that specialisation upon a sound scientific basis, and to avoid drifting into mere machines in controlling a limited section of trade or industry. Up to the present the policy of the trade associations has been almost universally to entrust the analytical work to trained chemists who have won the confidence of the public; and I think it will be a bad day for the trades concerned and for our profession if, as has been proposed in some

cases and adopted in others, analysts are appointed at fixed salaries, or inadequate fees, to undertake the work of the associations I have referred to.

The question of professional fees, and the remuneration of chemists in general, is a somewhat complicated one, and there does not seem to be any way in which one or the other can be standardised. With regard to the fees charged by practising analysts, I think it will be generally agreed that before the war the charges made, even by leading men, were in many directions quite inadequate. This was partly due to a keen competition amongst chemists for what little outside analytical work was required in the earlier days, when the analyses were required for private information or control purposes. In some cases the fees were cut by men who had been inadequately trained, but as often no very great degree of accuracy was required, and very little opportunity was afforded of checking the results, there was no elimination of the least capable, and the competition of these with the better trained men kept fees at a low level. In this way in many branches of work fees became more or less fixed, and by a general understanding they had to be accepted, especially by the younger members of the profession. Once fixed for any particular analysis it was very difficult for the individual to raise the fees without the risk of losing the work altogether. Thus it has happened, in my case at all events, that a very considerable part of my practice was carried on at very little profit to myself.

With the altered conditions brought about as the result of the war it became imperative that a revision of fees should be made, and even the most altruistic of us were compelled by the force of necessity to raise our fees. The Institute of Chemistry made inquiry into the matter, and in the February number of the *Journal and Proceedings of the Institute* for 1920, stated that, as the result of their investigations, "the Council are of opinion that Fellows and Associates are justified in making a corresponding increase in their fees for professional services." Although unable to recommend any definite rate of increase over pre-war rates, the Council of the Institute state that an increase of 50 per cent. cannot be regarded as excessive, while some practitioners have doubled their fees. This practically is the attitude which I myself have taken, readjusting the fees according to the work involved, but it is a matter of doubt whether the increases made in the fees cover the extra costs involved under present conditions.

With regard to chemists employed in whole-time engagements, I am afraid that in many instances the increases in remuneration have not been commensurate with the enhanced value of their services and the extra cost of living: but it is to be hoped that with the efflux of time and the stabilisation of the exchanges, the burdens at present borne will gradually be removed, and adjustments made which will remedy the present hardships.

During the past year your Council has worked assiduously, and, under the able guidance of our Secretaries, has kept the business of the Society and the interests of its members constantly in review.

One Committee, consisting of Messrs. L. Archbutt, W. J. A. Butterfield, G. Nevill Huntley, and G. Rudd Thompson, appointed by the Council to consider the desirability of making provision for supplies of standard chemical substances, has transmitted to the Council of the Institute of Chemistry a Report a summary of which appeared in the September number of the *ANALYST*.

The Committee is satisfied that there is a demand for Analytical Control Standards, at present being chiefly met by private enterprise and by the importation of the U.S.A. Bureau of Standards Samples, and that the demand appears reasonable for checking and educational, rather than commercial purposes.

The Committee recommend that the Society of Public Analysts should take the initiative in forming a representative Analytical Standards Committee, and the Council of the Institute of Chemistry have concurred with that recommendation, and have nominated Mr. F. H. Carr, Dr. J. T. Dunn, Mr. Lewis Eynon, and Mr. F. W. Harbord to act as its representatives.

That standards such as have been prepared or proposed would prove a great service, especially for educational purposes, scarcely admits of a doubt: but when it is suggested that these standards should be analysed side by side with commercial samples submitted for analysis, and that the fact should be stated on the certificate, I am very clearly of opinion that such use would be detrimental to the profession, and damage its prestige. In most cases the materials for which standards would be prepared would be commercial products, the analyses of which are almost invariably placed in the hands of experts, who are past-masters in the particular branch of analysis. To make any statement such as that suggested would be tantamount to an admission of incompetency.

And now, in conclusion, I have to express to our indefatigable and ever courteous Secretaries, Mr. P. A. Ellis Richards, and Mr. E. Richards Bolton, the deep debt of gratitude under which I have been placed by their many kindnesses and invaluable promptings, both with regard to procedure and policy, during the year now coming to an end.

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THE TIME FACTOR IN SAPONIFICATION.

By PERCIVAL J. FRYER, F.I.C.

(*Read at the Meeting, November, 1920.*)

VARIOUS conflicting statements have been made by observers with regard to the relative velocities of saponification of natural oils and fats under identical conditions of solvent, temperature, saponifying reagent, etc.

Thus, in the case of hydrolysis of castor bean ferment Urbain, Saigon and Feige came to the opinion that the saponification of various glycerides proceeds at the same rate (*Bull. Soc. Chim.*, **31**, 1194). Haller, on the other hand, was of opinion that glycerides of the lower fatty acids were more readily hydrolysed than those of the higher fatty acids (*Compt. rend.*, 1906, **143**, 660).

As far as the writer is aware, the only research which has been carried out using an alkali such as potassium hydroxide as a saponifying reagent for the natural oils and fats is that by Anderson and Brown (*Journ. Phys. Chem.*, 1916, **20**, 195-213), and it was mainly with a view to confirming or disproving the results obtained by those

investigators that the present research was carried out. There was also the possibility of basing an analytical test for the discrimination of oils and fats on the results obtained, in case individual oils and fats showed sufficient divergence for the purpose.

Anderson and Brown state that they find that the fats and oils investigated were all saponified at approximately the same rate, but they also conclude that the velocity increases slightly with the increase of the molecular weights of the glycerides. It is, however, fair to add, that they modified this statement by suggesting that such an apparent relationship may be due to experimental errors. Unfortunately, this comparison was based upon results with only four oils—namely, cottonseed, croton, olive, and castor—and although the velocity of saponification of butter fat was also investigated, apparently only one determination was made, and for some reason the result obtained was disregarded in arriving at their deductions. The method used by Anderson and Brown, was, in the author's opinion, unlikely to give results of sufficient accuracy on which to base conclusions as to the relative velocity of oils which resemble each other fairly closely. Thus, they did not weigh their oil, but measured it only, and no temperature is given at which the oil was pipetted. Further, it is not stated what means were taken to remove the residue of viscous oils, such as castor, which would remain in the pipette, unless special means were taken to flood the latter.

The conclusion arrived at by the writer as the result of the present investigation is that the exact opposite of the above relationship must be taken as correct—namely, that the velocity of saponification of oils and fats from the point of view of the amount of free alkali removed from the reacting solution is in inverse ratio to the saponification equivalent or to the mean molecular weight of fatty acids of the glycerides composing the natural oils and fats.

The statement is made in this form because previous investigators, including Anderson and Brown, have, in all cases, measured the velocity of saponification in terms of the free alkali removed from the solution by the glyceride. In terms of the weight of oil or fat employed, the results show that all oils and fats, and in all probability all esters, of whatever constitution, are saponified at approximately equal rates under the same conditions. Thus, using a given solvent and a given concentration of potassium hydroxide, the same percentage of the weight of an oil such as coconut is saponified in a given time as in the case of, say, rape oil, but in terms of the free alkali absorbed the former case shows much more rapid velocity of saponification than the latter. Incidentally, it may be here pointed out that the results obtained by Anderson and Brown with butter fat, which is the only oil or fat employed containing glycerides of low molecular weight, confirmed the author's work, and show a proportionately higher velocity of saponification, although, as previously stated, the authors appear to ignore this in their deductions.

In their investigation as to the nature of the reaction (which they prove to be bimolecular in character) they obtained comparative results with methyl acetate, and the figures given show that this ester also conforms to the rule stated above—namely, that the velocity of saponification is in inverse ratio to the molecular weight, from the point of view of the saponifying reagent, or, in terms of the weight of ester, is saponified at a rate uniform with all other esters.

In a recent investigation, Franck (*Seifen Fabr.*, 1920, 40, 293-4) ascertained the relative velocities of saponification of linseed oil, its glycol ester, and its ethyl ester, the two latter synthetically prepared, but found that there were small differences in the speed of the reaction, the relative velocities being in the order: (1) Linseed oil; (2) glycol ester; (3) ethyl ester.

Here, again, the relative velocity appears to be in the inverse order of the molecular weight.

The oils and fats employed in this investigation were typical of the various groups of oils and fats as follows:

Marine oils	...	...	...	...	Whale oil.
Drying oils	...	...	...	...	Linseed.
Semi-drying oils	...	...	...	...	Cotton.
Non-drying oils	{ Group I.				Rape.
	{ Group II.				Olive.
	{ Group III.				Castor.
Vegetable fats	{ Group I.				Cacao butter.
	{ Group II.				Palm oil.
	{ Group III.				Coconut oil.
Animal fats	{ Group I.				Tallow.
	{ Group II.				Butter fat.

The author's work confirmed that of previous investigators, in showing that the velocity was influenced very greatly by small differences in temperature. To such an extent is this the case that it appears very difficult to obtain reliable comparative results, unless all the oils investigated are saponified side by side, under identical conditions as to temperature. Thus, an increase of about 7-8° in temperature results in doubling the velocity of saponification. As regards the effect of the solvents on the velocity of saponification, the alcohols show an increased velocity in direct proportion to their molecular weights: thus the velocity as given in ethyl alcohol is over ten times that in methyl alcohol, and in amyl alcohol is about double that in ethyl alcohol.

If amyl alcohol be used, no other solvent need be employed for the oil or fat; the only oil which can be saponified in solution, using ethyl and methyl alcohols, is castor oil. The solvent employed in the author's investigations of the comparative velocities of the typical oils and fats was a mixture of ether and absolute alcohol, in which they all readily dissolved.

The velocity is also increased by an increase in the concentration of alkali, and the effect of this increase in alkali is less pronounced if water is present with the alcohol. This latter is obviously due to the different degree of ionisation of the potassium hydroxide.

LABORATORY TEST.

As a comparatively simple and rapid test, involving no heating or special apparatus, one on the following lines might be found of some utility.

A convenient quantity, say 2 grms., of the fat to be examined is weighed out into a flat-bottomed flask of, say, 100 c.c. capacity. This is dissolved in, say, 20 c.c. of ether and securely corked. The same weight of a pure ester (for convenience

amyl acetate) is weighed out and dissolved separately in the same volume of ether. To each is added an equal volume (say, 25 c.c.) of $\frac{N}{2}$ alcoholic potassium hydroxide solution (all the liquids should be at identical temperatures, and remain so); the two flasks are then securely corked and allowed to stand for, say, thirty minutes. At the end of this time an accurately measured excess of $\frac{N}{2}$ hydrochloric acid is run into each and the mixture back-titrated with $\frac{N}{2}$ alkali.

The mean molecular weight of the glycerides of the oil is then ascertained by comparison with the known molecular weight of amyl acetate, or the result may be expressed in terms of the saponification value, given that of amyl acetate as equivalent to 637.5.



IODIMETRIC DETERMINATION OF THE DIASTATIC POWER OF MALTS.

By JULIAN L. BAKER, F.I.C., AND HENRY F. EVERARD HULTON, F.I.C.

(*Read at the Meeting, February 2, 1921.*)

THE determination of diastase in malt cannot, as is well known, be made directly, but is always effected by measuring the capacity of the enzyme to produce sugar from starch. This reaction was first put on a scientific and quantitative basis by Kjeldahl (*Comptes rend. Trav. Lab., Carlsberg*, 1879, 1, 109-157), who formulated his well-known "law of proportionality" as follows:

"The relation between two malt extracts (and consequently between two malts) as regards their diastatic powers may be expressed by the copper oxide reducing powers which they exhibit when both act on the same quantity of starch at the same temperature, provided the copper oxide reducing power, $k=25-30$, is not exceeded."

Since it is customary in modern starch work to express reducing powers in terms of maltose (R), and not dextrose (k), it may be stated that $k\ 25-30=R$ (maltose) 40 to 48.

The method now in general use is that of C. J. Lintner (*Zeitsch. ges. Brauw.*, 1885, 8, 281), which is based on Kjeldahl's work, and involves the use of Lintner's soluble starch (*J. prakt. Chem.*, 1886, 2, 34, 378-394). By this method malt is said to have a diastatic capacity Lintner of 100 when 0.1 c.c. of 5 per cent. cold water extract of the malt, if allowed to act for one hour at 70° F. upon 10 c.c. of 2 per cent. soluble starch solution, produces enough maltose to reduce completely 5 c.c. of Fehling's solution.

The working details of the Lintner "tube" method (*loc. cit.*) are so well known that a description in this paper is unnecessary, as are also those of the modification devised by A. R. Ling and adopted by the Malt Analysis Committee of the Institute of Brewing (*J. Inst. Brew.*, 1906, 12, 6). The latter method has now been in general use in most laboratories associated with the fermentation industries for many years with entirely satisfactory results. The point of complete reduction of the Fehling's solution, even when using the thiocyanate indicator described by Ling and

Jones (ANALYST, 1908, 33, 161) may present, however, some difficulties to those who only occasionally determine the diastatic activity of materials, and we consider the process we are about to describe is easier, as the end point is indicated by the disappearance of the characteristic blue of starch iodide.

It is known that maltose is oxidised quantitatively to maltobionic acid by iodine in presence of caustic soda (Judd, ANALYST, 1920, 45, 224; Baker and Hulton, *Biochem. J.*, 1920, 14, 754). Another method thus becomes available for the determination of diastatic capacity, as the maltose formed by the action of the diastase can be determined iodimetrically. Miss Judd and we (*loc. cit.*) find that 1 gm. of anhydrous maltose requires 0.745 gm. of iodine for oxidation to maltobionic acid—*i.e.*, 1 c.c. of $\frac{N}{10}$ iodine is equivalent to 0.017 gm. of maltose, and 5 c.c. Fehling are equivalent to 2.4 c.c. $\frac{N}{10}$ iodine. In practice excess of iodine is employed, and the iodine used up is determined by titrating back with $\frac{N}{20}$ sodium thiosulphate in the usual manner. The excess of soluble starch remaining in the products of the action of diastase on starch serves as an indicator in the titration, and a sharp end point is obtainable with 0.05 c.c. of $\frac{N}{20}$ sodium thiosulphate.

In using the iodimetric method the preliminary stages should be carried out exactly as in the Ling modification—*i.e.*, 1, 2, or 3 c.c. of extract are allowed to act for one hour at 21° C. on 100 c.c. of 2 per cent. soluble starch. The action is stopped at the end of the one hour by adding 10 c.c. of $\frac{N}{10}$ caustic soda solution; the whole is then diluted to 200 c.c. For the titration 50 c.c. of the conversion products are placed in a wide-mouthed stoppered vessel; 20 c.c. of $\frac{N}{10}$ iodine solution are then added, followed by 30 c.c. of $\frac{N}{10}$ caustic soda, the whole allowed to stand for ten minutes at air temperature, then acidified with 4 c.c. of $\frac{N}{1}$ sulphuric acid, and the excess iodine found by titrating with $\frac{N}{20}$ sodium thiosulphate solution. If these conditions are adhered to, the diastatic capacity may then be found from the formula :

D.P. = $\frac{16.7 Y}{X}$, where Y = c.c. of $\frac{N}{10}$ iodine used up in oxidising the maltose, and X = c.c. of 5 per cent. malt extract originally taken for the conversion.

TABLE I.

R (Maltose Percentage on Starch).	$\frac{R}{N}$ (N = c.c. of Diastase Solution employed).	Percentage fall in Diastatic Power.
13	12.7	
24	12.0	5.5
34	11.7	7.9
40	11.5	9.4
45	11.0	13.4
61	10.2	20
68	6.5	50

To check the reducing power (R) of the conversion when 50 c.c. are used for the iodimetric determination as above, the value may be found from the formula

$R = Y \times 3.4$. From this it is evident that if Y exceeds 10, then R will exceed 34. How important it is to be sure that this limit is not exceeded will be seen from the figures in Table I., obtained from an ordinary pale malt, which show that at R 34 there is already a fall in the diastatic power of 8 per cent. as compared with the diastatic power found when the reducing power as maltose is not allowed to exceed 13.

The expression $\frac{R}{N}$ should be a constant if the maltose formed is a linear function of the quantity of diastase present, but this is never quite the case, although the departure is but slight in the earlier stages. Special stress is laid on this point, since it is often overlooked by writers on the subject (ANALYST, 1910, 35, 360; 1920, 45, 453).

The volume of conversion used (50 c.c.) for the titration may, of course, be halved, in which case 10 c.c. of $\frac{N}{10}$ iodine and 15 c.c. $\frac{N}{10}$ NaOH would be sufficient, and the necessary corrections must then be made in the formulæ.

In Table II. will be found the results of some determinations made by the Ling and iodine methods, a portion of the same conversion products being tested both with iodine and Fehling's solution.

TABLE II.

	R (Maltose per Cent.).	Diastatic Power.	
		Ling.	Iodine.
Pale Ale English malt ...	16	40.8	39.6
Pale Ale English malt ...	21	34.5	34.5
English Mild Ale malt ...	19	31.8	32.0
Californian malt ...	19	31.8	31.5
American malt ...	21	105.3	106.7
Green malt (5 days' germination)	34	55.5	56.0
Malted rye ...	9	87.0	86.7

It will be seen that a diastatic value is assigned to a sample of green malt. Doubt has been cast upon the applicability of the Kjeldahl law of proportionality to such material (Ling, *J. Inst. Brew.*, 1902, 8, 444), since it has been stated that the $\frac{R}{N}$ value (see Table I.) falls somewhat rapidly. We have not investigated this point in the case of green malt, but have found (*J. Soc. Chem. Ind.*, 1908, 27, 370) that both wheat and barley flour show a more marked fall in the value of this constant than is the case with barley malt. The fact that maltase occurs as an endocellular enzyme in green malt (Davis, *Biochem. J.*, 1916, 10, 31-48; also Daish, *ibid.*, 1916, 10, 56-76) may possibly account for this discrepancy since any formation of dextrose from maltose—if it occur—will upset the linear relationship of “apparent maltose” to “enzyme present” upon which the Kjeldahl law is based.

In conclusion, it may be stated that neither the Fehling nor the iodine method is accurate if any reducing sugar other than maltose be formed in the course of the conversion, as is the case, for example, with taka diastase, where both maltose and

dextrose are formed (ANALYST, 1914, 39, 312; 1920, 45, 141 and 453). Since both these sugars react with Fehling's solution and with iodine and the percentage amount of each sugar present varies with the stage of the conversion, it is evident that the reducing power alone of the solution cannot be a measure of the quantity of enzyme present.

The advantages claimed for the iodine method are the greater accuracy with which the iodine titration may be carried out as compared with Fehling's solution, the elimination of an external indicator, and that artificial light can be used for the final titration.

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NOTE.

The Editor desires to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

ZEISS BUTYRO-REFRACTOMETER—SETTING OF SCALE.

DURING the war it was impossible to obtain a standard liquid for setting the scale, but now a standard liquid paraffin of refractive index 1.474 at 15° C. is procurable. As 15° C. is not a convenient temperature for standardisation, readings were taken at other temperatures with the standard paraffin, the refractive index of which was kindly confirmed by Professor G. T. Morgan at the Birmingham University.

In the table below the figures under "A" were obtained with the liquid paraffin, the scale being set at 72.7 at 15° C., which is equivalent to refractive index 1.474; those under "B" with the same scale setting, and with the standard fluid obtained from Messrs. Zeiss about eight years ago. Fresh fluid was used for each reading, as continued heating may cause alteration in the reading.

The figures under "C" for 15°–30° C. are taken from the pamphlet supplied by that firm, whilst the other figures are calculated on the assumption that the alteration of values is in the same straight line.

C	A.	B.	C.
15°	72.7	78.2	77.3
20°	69.5	75.2	74.3
25°	66.5	72.1	71.2
30°	63.3	68.9	68.1
35°	60.5	65.8	65.1
40°	57.7	62.8	62.1
45°	55.0	60.0	59.1

The values under "B" and "C" should be identical, but there was a difference of nearly a unit. Therefore, a refraction obtained with an oil would be nearly a unit lower if the Zeiss fluid had been used for setting the scale than if the paraffin had been used. Whether these differences are due to the Zeiss liquid having deteriorated or to an original error in its composition, we are unable to say.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Detection of Beef Fat in Lard. Vitoux and C. F. Muttelet. (*Ann. Falsif.*, 1920, 13, 593-601.)—As shown by Bömer (*ANALYST*, 1913, 38, 204), lard contains α -palmitodistearin, m.-pt. 68.5° C., and beef fat β -palmitodistearin, m.-pt. 63.3° C. For the detection of beef fat in lard, the authors propose a method which depends on the crystallisation of a fraction from the fat under examination, the m.-pt. of this fraction showing which of the two above-mentioned glycerides predominates. Fifty grms. of the fat are dissolved in 50 c.c. of acetone, the mixture being heated slightly to aid solution, and then allowed to cool and, after one hour, the crystalline mass is collected on a filter. As much as possible of the liquid is removed by suction; the crystals are then dried over sulphuric acid and recrystallised from 50 c.c. of ether, and the new crystals collected and dried. In the case of pure lard the m.-pt. of the crystals lies between 62° and 65° C., whilst the crystals obtained from beef fat melt at about 58.5° C. If a portion of the crystals be saponified and the fatty acids liberated and separated, their m.-pt. will be about 58° C. for lard, and 55° C. for beef fat. For pure lard, the expression $2G - A$ is not less than 68; G is the m.-pt. of the crystals and A that of the fatty acids. W. P. S.

Prickly Pear Seed Oil. S. Lomanitz. (*J. Ind. Eng. Chem.*, 1920, 12, 1175.)—The seeds yielded 11 per cent. of a greenish-yellow, semi-drying oil, having the following characters: Specific gravity at 15.5° C., 0.9294; refractive index at 40° C., 1.46764; acid value, 3.09; saponification value, 189.5; iodine value, 116.3; Reichert-Meissl value, 2.8; insoluble fatty acids, 93.8 per cent. W. P. S.

Identification of Soya Bean Oil. C. A. Newhall. (*J. Ind. Eng. Chem.*, 1920, 12, 1174-1175.)—Five c.c. of the oil are shaken thoroughly with 5 c.c. of chloroform, a few drops of gum arabic solution, and 5 c.c. of 2 per cent. uranium nitrate or uranium acetate solution. Crude and refined soya bean oil yields a characteristic lemon-yellow emulsion, whilst earthenut oil, cottonseed oil, sesamé oil, rape oil, coconut oil, etc., give a white or slightly coloured emulsion. The test will detect the presence of 5 per cent. of soya bean oil in these oils. With linseed oil the test is not so sharp, since linseed oil gives a slightly brownish emulsion. The yellow emulsion is not obtained with bleached and deodorised soya bean oil or soya bean oil fatty acids. W. P. S.

Oils from Chinese Colza Seed (*Brassica campestris chinoleifera*). A. Viehoever, J. F. Clevenger, and C. O. Ewing (*Jour. Agric. Science*, 1920, 20, 117-139.)—The volatile oil was prepared by macerating the ground seed for two hours at 37° C., distilling it with steam, saturating the distillate with sodium chloride and extracting it with ether. The ethereal solution was dried by means of anhydrous sodium

sulphate, distilled to remove most of the ether, and exposed to air to allow the remaining ether to evaporate spontaneously. The residue obtained had a sp. gr. of 0.960 at 25° C., and distilled between 165° and 172° C. at 754 mm. Determination of the composition and constants of the thiourea and phenylthiourea compounds of the oil showed it to consist of crotonyl isothiocyanate. The fixed oil obtained by expression of the seeds had the following characteristics: Sp. gr. (25° C.) 0.9097; iodine value (Hanus) 100.3; saponification value 174; refractive index (25° C.) 1.4695; and Hehner value 96.1 per cent. The insoluble acids had a neutralisation value of 172.6, mean molecular weight 325, and iodine value 104. The percentage of liquid acids present was 75.82, and of solid acids 19.52, these having an iodine value of 55.2. The seeds contain 0.4 to 0.6 per cent. of volatile oil, and from 40 to 50 per cent. of fixed oil. The paper contains a botanical description of Chinese colza and its seeds, and is illustrated by numerous photographs. T. J. W.

Orange Vinegar: Its Manufacture and Composition. H. D. Poore. (*J. Ind. Eng. Chem.*, 1920, **12**, 1176–1179.)—Brief descriptions are given of the two processes used to produce orange vinegar, one in a small way by the barrel or roller method, and the other on a larger scale by the well-known acetifier method. The composition of the orange-juice, the fermented juice and vinegar prepared from the same by the acetifier method, is shown in the following table, the figures representing grms. per 100 c.c., except for the alcohol, which is per cent. by volume:

	Fresh Juice.	Fermented Juice.	Vinegar.
Total acid, as citric acid ...	1.14	1.13	—
Total acid, as acetic acid ...	—	—	4.74
Fixed acid, as citric acid ...	1.12	1.03	1.07
Volatile acid, as acetic acid ...	—	0.09	3.74
Total solids ...	13.39	3.64	4.34
Total sugars, as invert sugar ...	9.91	—	0.20
Alcohol ...	—	5.73	0.43
Total ash ...	—	0.48	0.61
Water-insoluble ash ...	—	0.10	0.17
Water-soluble ash ...	—	0.38	0.44
Alkalinity of soluble ash (c.c. of $\frac{N}{10}$ acid per 100 c.c.) ...	—	51.20	55.20

Orange vinegar resembles apple vinegar in colour, odour, and taste; the total acidity of the two classes of vinegar is about the same, but orange vinegar contains more fixed acid. Orange vinegars made by the barrel method are rather higher in acidity. W. P. S.

Estimation of the Extract of Coloured Malts. C. G. Matthews. (*J. Inst. Brewing*, 1921, **27**, 22–25.)—The following method is advocated by the author for the analysis of “crystal” and black malts in preference to that adopted by the Institute of Brewing, Malt Analysis Committee (*J. Inst. Brewing*, 1906, **12**, 6), on the

grounds that the latter method requires much time, a large quantity of standard malt, and tends to yield low results, besides giving variable extracts in the hands of different analysts: Forty grms. of standard malt, having a diastatic power between 35° and 45° Lintner, are ground in a Seck mill set to 25° and mixed with 10 grms. of the malt under examination ground in a similar manner (black malts may also be ground in a coffee-mill without any effect upon the final result). The mixture is mashed, cooled, and filtered according to the Committee's method, and the extract and colour determined in the usual manner by appropriate calculation. The use of a standard malt having a diastatic power above 55° Lintner yields abnormal results by both methods. Four tables giving the results of experiments obtained with different malts and with various modifications of method indicate that the procedure recommended by the author is reliable.

T. J. W.

Bracken Rhizomes. J. Hendrick. (*Chem. News*, 1921, **122**, 5-7.)—Bracken rhizomes resemble potatoes in composition, but the "soluble carbohydrates" of the former consist largely of substances of undetermined nature. Meal prepared by drying the rhizomes in a kiln and grinding was found to have the following composition: Moisture, 9.81; ether extract, 2.54; albuminoids, 9.56 (containing total nitrogen, 1.53, and protein nitrogen, 1.49); soluble carbohydrates, 41.26; fibre, 20.49; and ash, 16.34 (containing 11.26 per cent. of siliceous matter). Preliminary feeding experiments upon animals are described which indicate that the rhizomes may be of use for maintenance purposes, but are not very palatable to certain classes of stock.

T. J. W.

New Reagent for Bitter Lactarius and Russula Species. Barlot. (*Comptes rend.*, 1921, **172**, 87-89.)—With various fungi, methyl (or other) alcoholic solutions of methyl chloroantimonate containing 20-30 per cent. of chlorine give colorations, which are especially pronounced with *Lactarius* and *Russula*, the bitter species of these two genera behaving similarly. Thus, any part of the carpophore of *Russula emetica* or *Lactarius piperatus* assumes an intense leaden-grey coloration after contact for a few seconds with the reagent. The bitter principles of these two species were isolated, and appear to be identical. All the bitter *Russulae* give a similar coloration with varying intensity, whereas the edible species either produce no colour or else gradually give rise to a transient blue coloration. *Russula rosea* forms an exception, yielding immediately an intense grass-green coloration.

T. H. P.

Estimation of Acetic Acid in Pyroligneous Acid. V. E. Grotlisch. (*J. Ind. Eng. Chem.*, 1920, **12**, 1183-1186.)—The method consists in distillation of the acetic acid with xylene, neutralisation of the distillate, and oxidation of the distilled impurities (tars, phenols, aldehydes, etc.) with permanganate; after removal of the oxidised impurities, the residual sodium acetate is distilled with phosphoric acid as usual. Ten c.c. of the sample are distilled with 120 c.c. of xylene (pumice-stone being added), until the distillate measures about 75 c.c., consisting of 10 c.c. of an aqueous layer and 65 c.c. of xylene; the distillate is diluted with water and titrated with $\frac{N}{2}$ sodium

hydroxide solution, phenolphthalein (in methyl alcohol) being used as indicator. The xylene is then separated, and the aqueous portion is evaporated to 70 c.c.; 100 c.c. of water are added, the mixture again evaporated, and these operations are once more repeated. The residual solution is rendered slightly alkaline and potassium permanganate is added, the mixture heated on a water-bath for several hours, then cooled, diluted to 300 c.c. and filtered; 200 c.c. of the filtrate are evaporated to 75 c.c., cooled, a few drops of sulphuric acid and hydrogen peroxide are added, followed by a slight excess of barium hydroxide solution; the mixture is evaporated to a small volume and filtered, the filtrate is distilled with the addition of phosphoric acid, and the acetic acid in the distillate is titrated.

W. P. S.

Review of Tests for Methyl Salicylate in Gaultheria and Birch Oils.

C. H. LaWall. (*Amer. J. Pharm.*, 1920, **92**, 891-895.)—Since oil of gaultheria contains 99 per cent., and oil of birch over 99·5 per cent., of methyl salicylate, it is improbable that any infallible method exists for detecting the presence of one of these oils, even in large proportion, in the other. The only likely distinguishing reactions are colour reactions, which are usually uncertain, and, when used for quantitative purposes, rarely accurate. These observations apply to the suggestion to treat the oils with reagents for furfuraldehyde; with aniline acetate, for example, gaultheria oil gives an immediate intense red coloration, birch oil a pronounced red coloration, more slow to appear, and methyl salicylate a negligible reaction. With mixtures, however, such a test is valueless, and is easily defeated by the addition of a small proportion of furfuraldehyde to the methyl salicylate used as adulterant. The negative optical rotation required of gaultheria oil by the U.S. Pharmacopœia does not appear to be a distinctive character of this oil.

T. H. P.

Estimation of Hydrocyanic Acid. R. L. Morris. (*Amer. J. Pharm.*, 1920, **92**, 908-916.)—The results of the author's experiments show that, when properly conducted, Liebig's method for estimating cyanides by titrating the alkaline cyanide solution with standard silver solution gives quite satisfactory results. Deficiency of sodium hydroxide in the solution leads to low results, whilst any great excess of the alkali delays the end point and thus causes high results. The uncertainty occurring when too much alkali is employed may be avoided by replacing the sodium hydroxide by borax solution as proposed by Guérin (1906). Comparison of the B.P. process with that of the U.S.P. and the French Codex shows that the first apparently prescribes too little potassium iodide; if, however, the amount of the latter be increased two or three times the process agrees well with the gravimetric results.

T. H. P.

Content of Aconitine in some Samples of Tincture of Aconite Leaves.

A. Richaud. (*J. Pharm. Chim.*, 1921, **23**, 15-16.)—Estimation by the silico-tungstic acid method of the aconitine in four samples of tincture of aconite leaves obtained from four of the best-known druggists in Paris gave the respective amounts per grm. as: 0·409, 0·172, 0·139, and 0·167 grm.

T. H. P.

Method for the Assay of Aconite Preparations. E. J. Chappel and N. L. Allport. (*Amer. J. Pharm.*, 1920, **92**, 922-923.)—In the assay of tincture and liniment of aconite, first made official in the 1914 edition of the British Pharmacopœia, the analyst is left to base his process upon that described for the root, the liquid preparation being evaporated to dryness at a temperature not exceeding 60° C., and the solid residue then dissolved in $\frac{N}{50}$ sulphuric acid. Since filtration of the resulting solution is often very slow, and since, also, extraction of the unfiltered acid liquid with ether yields troublesome emulsions, the authors have tried petroleum spirit with good results. Fifteen c.c. of the liniment, or 100 c.c. of the tincture, are evaporated at a low temperature to remove the bulk of the alcohol, the residue treated with 5 c.c. of 10 per cent. sulphuric acid solution diluted with 20 c.c. of water and the whole transferred to a separating funnel with the help of a glass rod to break the separated resin; the dish is then rinsed carefully with a little water. About 20 c.c. of petroleum spirit (b.-pt. 40°-60° C.) are introduced into the funnel and the latter shaken, rapid separation occurring; the aqueous liquor is drawn off and again shaken with petroleum spirit, the two petroleum liquors being then mixed and washed twice with water, and the washings added to the acid liquid, which is then rendered alkaline with ammonia and extracted four times with ether. The ethereal extracts are washed successively with the same portion of water, after which they are run into a flask and evaporated to dryness. The alkaloidal residue is dried at a low temperature to ensure removal of ammonia, and then titrated in the usual manner. The process may be applied to the assay of the root by first preparing a tincture as directed in the Pharmacopœia. The results furnished by this modified method are to all intents and purposes identical with those given by the more lengthy B.-P. process. T. H. P.

Chemistry of Digitalis. H. C. Hamilton. (*J. Ind. Eng. Chem.*, 1920, **12**, 1180-1181.)—The author has isolated two active principles from digitalis, one substance being soluble in chloroform and the other insoluble; the insoluble substance is more active, physiologically, and there appears to be a tendency for the soluble substance to become less soluble on repeated applications of the solvent. Attention is directed to the difficulty of separating chloroform-soluble constituents from those insoluble in chloroform and of proving the latter to be less well adapted to therapeutical use. W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Colostrum. C. Porchet and L. Panisset. (*Comptes rend.*, 1921, **172**, 181-183.)—Colostrum is considered to be a residue of milk which has been formed previously in the udder and is the result of leucocytic action. The lactose is re-absorbed rapidly, proteins more slowly, and the fat remains. These changes may be observed when milk is injected into the peritoneal region of guinea-pigs, but in this case the products are not affected by the continual secretion of fresh quantities of milk as occurs in the udder. The residual fat globules, particularly the smaller ones, combine with the leucocytes yielding clusters of cells known as Donné's "corps granuleux," which are characteristic of colostrum. W. P. S.

An Epidemic of Ropy Milk. H. A. Harding and M. J. Prucha. (*Univ. of Illinois, Agric. Exper. Stat., Bull. No. 228, June, 1920.*)—During the summer of 1919 the attention of the authors was drawn to a severe outbreak of ropiness in milk which assumed the character of an epidemic, as it involved more than one hundred farms. The cause was not definitely ascertained, but the organisms causing the ropiness were found in the water supply, and this infected the dairy utensils. Disinfection of the latter and of the cowsheds usually results in the disappearance of the trouble. The organisms are destroyed at 130° F., and if milk be pasteurised for thirty minutes at this temperature it will not become ropy, although it may have been infected.

W. P. S.

Influence of the Diet of the Cow upon the Nutritive and Anti-scorbutic Properties of the Cow's Milk. R. A. Dutcher, C. H. Eckles, C. D. Dahle, S. W. Mead, and O. G. Schaefer. (*J. Biol. Chem., 1920, 45, 119-132.*)—From feeding experiments upon a Jersey and a Holstein cow, described in detail, the conclusion is drawn that the vitamin content of cow's milk depends upon the proportion of vitamin in the fodder. It was found that 20 c.c. of summer milk were superior in nutritive value and anti-scorbutic power to 60 c.c. of winter milk.

Relation of Fodder to the Anti-scorbutic Power and Salt-Content of Milk. A. F. Hess, L. J. Unger, and G. C. Supplee. (*J. Biol. Chem., 1920, 45, 229-235.*)—Five Holstein cows were fed for three weeks on dry fodder and then placed on pasture, with the addition of concentrated food, for three weeks. The milks at the close of the two periods differed to a pronounced extent in their content of anti-scorbutic vitamins, as indicated by the results of feeding experiments on guinea-pigs. The composition of the milk as affected by the fodder is shown by the following percentage results:

	Water.	Total Solids.	Fat.	Total Proteins.	Cascin.	Albu- min.	Leci- thin.	Lac- tose.	Citric Acid.	Ash.	P ₂ O ₅ .
Anti-scorbutic-free fodder	88.38	11.62	3.37	2.82	2.28	0.42	0.069	4.73	0.08	0.606	0.158
Pasture	88.19	11.81	3.44	2.93	2.32	0.49	0.7	4.56	0.13	0.670	0.190

In view of the pronounced variation in the amount of citric acid in the two milks, the conclusion is drawn that normal figures which have been established without regard to the fodder of the cows must be regarded as incomplete. The results also show the danger of setting up rigid standards for milk constituents without full data as to the food of the animals.

Growth promoting Factors in Lower Vegetable Organisms. P. Goy. (*Comptes rend., 1921, 172, 242-244.*)—Yeasts, mould fungi, and bacteria contain a growth-promoting substance which is distinct from the vitamins A and B in higher plants. This substance, as isolated from a culture of *Mucor mucedo* by extraction with ether, and purified by crystallisation, is a carbon compound free from phosphorus and nitrogen. It melts at about 175° C., and is not precipitated by phosphotungstic

acid. It only exerts its growth-promoting function after its solution has been heated to 85°-90° C., and loses its power at about 168°-170° C. Analogous results have been obtained by means of extracts of vegetable tissues, only the juice of the lemon and orange being active without previous heating. This substance has also been found in decorticated rice. It is not indispensable to the life of lower vegetable organisms, but has a pronounced stimulative action upon their growth.

Globulin from the Cohune Nut. C. O. Johns and C. E. F. Gersdorff. (*J. Biol. Chem.*, 1920, **45**, 57-67.)—Cohune nut globulin has been isolated by dialysis, and found to contain all the basic amino acids known to exist in proteins, being particularly rich in arginine and lysine. Tryptophane is also present. Cohune nut globulin closely resembles coconut globulin in composition.

Detection of Castor Oil Seeds in Feeding Cake. C. Brioux and M. Guerbet. (*Ann. Falsif.*, 1920, **13**, 150-160.)—The presence of castor seed as an impurity in oil cake may be detected by microscopical examination of the residue, and particularly the black particles left after repeated extraction of the powdered cake with boiling 2 per cent. potassium hydroxide solution. The distinctive appearance of the husk of the castor seed may be readily recognised. Ricin, the blood-coagulating toxin of castor seeds, may be isolated by treating 30 grms. of the powdered cake for several hours with 150 c.c. of 0.5 per cent. sodium chloride solution, and 5 c.c. of xylene, filtering the extract, heating it at 70° C. for an hour, and again filtering it. The new filtrate is saturated with ammonium sulphate, the resulting precipitate washed with 20 drops of water and dissolved in a 0.9 per cent. solution of sodium chloride at 37° C., and the solution filtered. On treating defibrinated blood with the filtrate, coagulation will take place within ten minutes if the original cake contained 2 per cent. or more of castor seeds. Cake containing even less than this amount of castor seed is poisonous to animals.

Colorimetric Estimation of Tryptophane. O. Fürth and F. Lieben. (*Biochem. Zeitsch.*, 1920, **109**, 124-152.)—Voisenet's reaction has been adapted to the estimation of tryptophan in proteins, etc. Two c.c. of the solution are treated with a drop of 2 per cent. formaldehyde solution and about 15 c.c. of concentrated hydrochloric acid. After about ten minutes 10 to 12 drops of a 0.05 per cent. solution of sodium nitrite are added, and then sufficient strong hydrochloric acid to make 20 c.c. in all. The violet coloration which appears after the liquid has stood for a short time is compared with that given under the same conditions by a 0.1 per cent. solution of tryptophane. The amounts of tryptophane thus estimated in various proteins are recorded, and it is shown that the method is also applicable in the case of insoluble and coagulated proteins. In test estimations the method showed an error of about 10 per cent.

Creatinine and Creatine in the Blood. C. C. Wang and M. L. Dentler. (*J. Biol. Chem.*, 1920, **45**, 237-243.)—The creatinine and creatine in the blood of twenty-four normal women was estimated by the method of Folin and Wu (*J. Biol.*

Chem., 1919, 38, 81.)—The amounts of creatinine ranged from 0.96 to 1.65 mgrms. (average 1.30 mgrms.) per 100 c.c., and thus did not support the suggestion that blood creatinine is lower in women. The creatine varied from 2.23 to 4.65 mgrms. per 100 c.c., which is slightly lower than the recorded range. The accuracy of creatinine estimations is affected by excessive sugar diet, owing to the additional colour caused by the dextrose.

Estimation of Chlorides in Blood. A. S. Wetmore. (*J. Biol. Chem.*, 1920, 45, 113-118.)—The method is based on the precipitation of protein by copper hydroxide, followed by precipitation of the oxalate and most, if not all, of the phosphate by excess of calcium hydroxide, and volumetric estimation of the chlorides: Two c.c. of the plasma (or 2 c.c. of blood) are diluted with 15 c.c. of water (or 10 c.c. in the case of blood), and treated with 4 c.c. (or 8 c.c.) of a 1.5 per cent. solution of copper sulphate and 10 c.c. (or 20 c.c.) of $\frac{N}{10}$ sodium hydroxide solution, and the flask heated for about a minute in boiling water, being meanwhile rotated. After cooling the liquid is made up to 50 c.c., thoroughly shaken and filtered, and 0.5 gm. of powdered calcium hydroxide is added to about 35 c.c. of the filtrate, which is then filtered after standing for one minute. The filtrate (25 c.c.) is treated with 5 c.c. of standard silver nitrate solution (AgNO_3 7.2653 grms., concentrated nitric acid 150 c.c., per litre) (1 c.c. = 2.5 mgrms. NaCl), and 2 c.c. of a 10 per cent. solution of ferrous ammonium sulphate, and the excess of silver nitrate titrated with standard potassium thiocyanate solution (1.6 grms. per 800 c.c. diluted until 12.5 c.c. react with 5 c.c. of the silver nitrate solution—i.e., 1 c.c. of thiocyanate solution = 1.0 mgrm. of sodium chloride).

New Biological Reaction for Blood. R. Guyot. (*Ann. Chim. Anal.*, 1921, [ii.], 3, 27-28.)—The reagent employed consists of *Boletus* tincture, prepared by heating slices of *Boletus cyanescens* in water until the latter boils, and then macerating them for forty-eight hours in 60 per cent. alcohol. This liquid soon turns blue, but becomes colourless when exposed to sunlight; it is then filtered. The reaction is carried out as follows: Two c.c. of a mixture of 2 drops of blood with 50 c.c. of distilled water are placed in a test-tube, into which are pipetted, carefully and without mixing with the liquid, a little of the above tincture, and then one or two drops of hydrogen peroxide solution. A greenish coloration soon forms at the surface of contact of the two liquid layers and afterwards changes to blue. The same reaction is given with hæmoglobin solution and with fresh hæmoglobinuric urines, but it is not given by blood treated with carbon monoxide; in the case of ordinary blood it becomes attenuated or disappears as the hæmoglobin molecule changes. The reagent is not specific for blood, but is applicable to all oxydases, including that of *Boletus* itself.

T. H. P.

Volumetric Method of Estimating Reducing Sugars in Blood and Pathological Fluids. A. Jonescu and V. Vargolici. (*Bull. Soc. Chim. România*, 1920, 11, 102-106.)—The method is a modification of one previously described (ANALYST, 1920, 45, 339), based on the reduction of ferricyanide to ferrocyanide by

reducing sugars. From 1 to 10 c.c. of the sugar solution is added to an excess of potassium ferricyanide solution (3 to 4 c.c.), diluted with 5 c.c. of water, and the liquid is then boiled for one minute, and after cooling is diluted with 100 c.c. of water and acidified with sulphuric acid (20 per cent.). The ferrocyanide formed is then titrated with permanganate solution (10.6 grms. per litre), which is conveniently standardised against a known solution of dextrose, and adjusted so that each c.c. corresponds to 0.001 grm. of dextrose. Tests with solutions of reducing sugar at concentrations of 0.1 to 1.0 per cent. gave accurate results. Trichloroacetic acid solution (20 per cent.) was used as a defæcating agent in the proportion of 1 : 1 for blood and 1 : 4 for pathological fluid, the filtered solution being neutralised with sodium hydroxide before estimating the reducing sugars. R. G. P.

Separation of Ptomaines and Alkaloids in Toxicological Analysis.

A. Jonescu. (*Bull. Soc. Chim. România*, 1920, 11, 82-89).—Experiments on the action of various enzymes upon morphine, strychnine, and conine, and upon cadaveric ptomaines prepared by Dragendorff's method, showed that the hydrolytic enzymes, invertin and amylase, have no action on ptomaines, and that pepsin has only a weak action, lessening the reducing power of ptomaines without influencing their alkaloidal character. The oxidising enzymes, tyrosinase and hæmoglobin, have a definite action on ptomaines, and remove their reducing powers without affecting their alkaloidal character. Enzymes did not cause any important change in the alkaloids used in the experiment; even morphine, which, according to Bourquelot and Bougault, is greatly affected by tyrosinase, was only slightly modified under the experimental conditions. The retention or disappearance of the reducing power of a supposed alkaloidal residue in the presence of hæmoglobin may therefore serve to distinguish its nature. It is suggested that other enzymes and different conditions of experiment may destroy both the reducing power and the alkaloidal character of ptomaines. R. G. P.

Bacteria in Non-Alcoholic Drinks. L. Gershenfeld. (*Amer. J. Pharm.*, 1920, 92, 803-806).—The author has examined various manufactories in which non-alcoholic beverages are prepared, and concludes that in most cases the water supply is satisfactory, and that bacterial contamination is due to careless washing of containers or the use of infected flavouring syrups. Beverages prepared under clear conditions are lower in bacterial content than the water used in their preparation. Out of fifteen samples of non-alcoholic beverages six showed the presence of *B. coli* in 10 c.c. portions, whilst one sample, although free from this organism, contained another sewage bacterium, *B. Welchii*. Total bacterial counts obtained on agar, when incubated for forty-eight hours at 37° C., gave two samples with less than 100 per c.c., three less than 300 per c.c., three ranging between 500 and 1,000 per c.c., whilst the remaining seven yielded above 1,000 bacteria per c.c. Nearly all samples gave a somewhat higher count at 20° C. than at 37° C. Among the organisms identified were *B. coli*, *B. Welchii*, *B. cloacæ*, *B. subtilis*, *B. mycoides*, *B. mesentericus vulgatus*, and various streptococci, diphtheroids, and streptothrices. It is suggested that more attention should be given to this problem from a sanitary point of view.

T. J. W.

Bacteriology of Canned Olives. S. A. Koser. (*J. Agric. Research*, 1920, 20, 375-379.)—Recent outbreaks of botulism traced to the consumption of ripe olives led to a bacteriological investigation of nearly five hundred containers of preserved olives. The cans or jars were opened under aseptic conditions, and from 1.5 to 2 c.c. of the liquor was removed, 0.5 c.c. of this being spread upon a dextrose-agar slope, and the remainder added to a tube containing dextrose-beef broth under oil, the cultures being then incubated at 37° C. One hundred and ninety-nine of the containers showed the presence of living organisms, and seven of the spoiled glass jars contained *B. botulinus*. In most of the samples of normal appearance the contents were sterile, but a small proportion yielded cultures of aerobic bacilli, cocci, or dormant members of the coli group. The majority of organisms identified belonged to the coli group, but members of fourteen other types were recognised. A report referring particularly to *B. botulinus* has already been made. (See *J. Amer. Med. Assoc.*, 74, 1220, 1920.) T. J. W.

Germicidal Value of Some Chlorine Disinfectants. F. W. Tilley. (*J. Agric. Research*, 1920, 20, 85-110.)—A detailed account is given of numerous experiments made to ascertain the antiseptic value of chloramine T, Dakin's] solution, eusol, and chlorine water upon various bacteria, and determinations of the carbolic acid coefficients were made by the Rideal-Walker method. The results show that with comparisons based upon the available chlorine in each antiseptic chloramine T is more efficient against *Staphylococcus aureus*, approximately equal in value against *B. typhosus* and much less efficient than the other compounds against *B. pyocyaneus*. *B. tuberculosis* is unaffected by any of these compounds when in a concentration of 1 in 200 of available chlorine. None of these substances has such a rapid action upon anthrax spores as is generally believed, and several days may be required to secure complete sterilisation with concentrations of 1 per cent. The addition of ammonia to chlorine or hypochlorite solutions greatly increases their antiseptic properties and tends to prevent their deterioration in the presence of organic matter. T. J. W.

WATER ANALYSIS.

Detection of Indican as a Criterion of the Purity of Water. A. Jolles. (*Ber. Deut. Pharm. Ges.*, 1920, 30, 421-442.)—From 3 to 4 litres of the water are evaporated to 250 c.c., and any nitrite present removed by the addition of 3 grms. of ferrous ammonium sulphate for each 0.1 gm. of nitrite in a litre of the sample. The liquid is then concentrated to 10 c.c. and filtered, and the filtrate tested with 1 c.c. of a 5 per cent. alcoholic solution of thymol or α -naphthol and 10 c.c. of concentrated hydrochloric acid containing 5 grms. of ferric chloride per litre. If the original water contained indican (potassium indoxyl sulphate) a coloration due to the formation of indolignone will be produced after the mixture has been left for fifteen minutes, with frequent shaking. Any colouring matter thus formed is extracted by shaking the liquid with 4 c.c. of chloroform, and if a reddish or bluish violet extract be obtained, the presence of indican is definitely indicated. The detection of even a trace of indican affords a proof that the water was contaminated with animal excreta.

Substances Dissolved in Rain and Snow. W. A. Moore and G. Browning. (*Chem. News*, 1921, 122, 51-52.)—The rain which fell between May and August, 1920, at Mount Vernon, Iowa, eighteen miles from the nearest manufacturing town, frequently contained traces of phosphates and sulphates, but was free from chlorides. Carbon dioxide was sometimes present, especially when the wind was from the manufacturing centre. Two precipitations on the same day contained respectively 0.001 and 0.0009 parts of nitrites, and 0.3 and 0.005 parts of nitrates per million, and a similar decrease was always observed in the rain following the rainfall of a previous day. The average rain of the summer months contained 0.28 part of ammonia, 0.35 part of nitrate, and 0.23 part of carbon dioxide (*cf.* ANALYST, 1914, 39, 190, 501).

ORGANIC ANALYSIS.

Estimation of Carbon Dioxide in Coal. F. S. Sinnatt and W. Harrison. (*Lancs. and Cheshire Coal Research Assoc., Bull.* No. 7, 1920, 15 pp.)—The apparatus devised for the estimation of carbon dioxide in carbonates (ANALYST, 1913, 38, 136) has also been found suitable for coal. From 0.5 to 5 grms. of the coal pulverised to pass through a $\frac{1}{80}$ -mesh sieve are introduced into the decomposition flask B (see diagram, *loc. cit.*), and covered with twice its volume (5 to 15 c.c.) of water, and the contents of the flask gently boiled for about twenty minutes to expel occluded gases. The flask is then connected with the apparatus, the flask E of which has previously been exhausted by means of an ordinary water pump, the carbon dioxide in the flask being thereby reduced to a negligible amount. Hydrochloric acid (5 per cent.) or dilute phosphoric acid (1 : 4) is allowed to run on to the coal from the cup L, and air drawn through the apparatus at a constant speed. After about ten minutes the decomposition flask is gently heated and its contents kept at 50° C. for thirty minutes, and finally boiled for ten minutes. Air is meanwhile swept through the flask at the rate of about 2 litres per hour, and the evolved carbon dioxide is collected in the flask E and estimated by means of standard barium hydroxide solution, as in Pettenkofer's method. The following amounts of carbon dioxide were found in Lancashire coals from which the white partings (ankerites) had not been separated: Mountain mine, 0.50; Arley, 0.18; Ravine, 0.36; Abnormal sample I., 6.85; Abnormal sample II., 18.32; Pemberton, 2 feet, 0.72; Garswood, 9 feet, 0.44; and Hoo cancell, 1.84 per cent.

Detection of Oils other than Linseed Oil in Paints by Means of the Hexabromide Test. H. Bailey and W. D. Baldisiefen. (*J. Ind. Eng. Chem.*, 1920, 12, 1189-1194.)—The following method gives more concordant results than some of the older methods: One grm. of the linseed oil fatty acids, prepared in a current of carbon dioxide, is treated with 25 c.c. of ether previously saturated with linolenic hexabromide at 0° C., cooled to about -10° C. in a bath of ice and hydrochloric acid, and brominated with a mixture of 5 c.c. of bromine and 25 c.c. of glacial acetic acid, which is added at the rate of one to two drops per second, with shaking after each addition, until the solution becomes permanently orange. The tube is left for sixteen hours in an ice-box, the precipitate then broken up, the tube centri-

fuged and again cooled in an ice-bath, and the ether decanted. The precipitate is washed with successive portions of 20 c.c. of the chilled saturated ether, and is finally dried for fifteen minutes at 60° C. under a reduced pressure of 30 to 40 mm. Seven samples of pure linseed oil fatty acids yielded from 41.1 to 44.1 per cent. of hexabromide, whilst commercial "boiled" linseed oil prepared without much heating (iodine value 173.8) yielded 42.6 per cent. Five samples of genuine soya bean oil yielded from 4.2 to 7.3 per cent. of insoluble bromides, whilst menhaden oil gave 35.9 per cent. Taking the average hexabromide value of linseed oil fatty acids as 42, and that of pure soya bean oil fatty acids as 6, it was possible to estimate fairly closely the proportion of linseed oil in mixtures of the two oils. Fish oils yield octobromides insoluble in ether, but these may be readily separated from the hexabromides of vegetable oils by their insolubility in warm chloroform. In the examination of samples of paints the conclusion may be drawn that when the oil, after a correction has been made for the unsaponifiable matter (about 1 per cent. in linseed oil), gives a hexabromide value of 39 or less, the presence of oils, other than linseed oil, is indicated. The changes that take place when linseed oil is oxidised in the air at high temperatures (400° F.) cause a decrease in both the hexabromide and iodine values, but not in the same proportion, and preliminary experiments have indicated that it may be possible to establish a relationship between the two values which will afford the means of distinguishing oxidised linseed oil from mixtures of such oil with other oils.

Disodium Hydrogen Phosphate as a Catalyst for the Oxidation of Dextrose with Hydrogen Peroxide. E. J. Witzemann. (*J. Biol. Chem.*, 1920, 45, 1-22.)—The author has determined the effect of various substances and other factors upon the oxidation of dextrose by hydrogen peroxide, and has given a detailed discussion of the results obtained. No definite method appears to have been worked out for the estimation of dextrose, but it is shown that this sugar may be quantitatively oxidised to carbon dioxide by the action of hydrogen peroxide in a solution containing a mixture of monosodium and disodium hydrogen phosphates. Since the carbon dioxide is evolved continuously during the course of the reaction, the phosphate mixture may be used repeatedly for additional quantities of dextrose, thus resembling a catalyst in its action. The phosphate mixture behaves only as a peroxidase, and does not catalyse the action of atmospheric oxygen. The action observed does not depend only upon the decomposition of hydrogen peroxide by the phosphate, since other substances more effective in decomposing the peroxide diminish the oxidation of dextrose. Sodium carbonate and bicarbonate, when in the same molecular proportions as the two phosphates, do not cause the oxidation of dextrose under similar conditions, and free alkali has no influence upon the oxidation. It is of interest biologically that disodium hydrogen phosphate is the only chemical substance known which is necessary to organic life processes, and which is capable of catalysing the quantitative oxidation of dextrose to carbon dioxide. T. J. W.

Preparation of Hydrazines. L. Thompson. (*Chem. News*, 1921, 122, 40-43.)—As a reduction agent in the preparation of hydrazines, sodium hydrosulphite

is superior to bisulphites or stannous salts, as its action is more energetic and promotes better yields. Phenylhydrazine hydrochloride may be prepared directly by reduction of benzene diazonium chloride with sodium hydrosulphite in presence of hydrochloric acid: $C_6H_5N_2Cl + 2Na_2S_2O_4 + 4H_2O = C_6H_5NH.NH_2.HCl + 4NaHSO_3$. If solid sodium hydrosulphite be used, the reaction does not take place quantitatively, a small quantity of a red compound, presumably azobenzene *p*-hydrazine-sulphonic acid, being produced. With only one molecule of the hydrosulphite, sodium phenylhydrazine sulphonate is first formed; on boiling with concentrated hydrochloric acid and cooling to 0° C., an 85 per cent. yield of phenylhydrazine hydrochloride is obtained. Hydrazine compounds are employed in the estimation of aldehydes and ketones, especially carbohydrates; *p*-nitrophenylhydrazine is a suitable reagent for the identification of oxycellulose.

W. J. W.

Examination of Wood Cellulose. F. Lenze, B. Pleus, and J. Müller. (*J. prakt. Chem.*, 1920-1921, 101, 213-264.)—Digestion of cellulose with 17 per cent. sodium hydroxide solution, for the separation of normal cellulose, effects solution of its other constituents, such as oxycellulose, hydrocellulose, cellulose dextrin, hemi-celluloses, pectin, lignin, resins, and fats; for the determination of these the solution is first treated with acids and alcohol. For the estimation of oxycellulose, 10 grms. of air-dried cellulose are treated with 100 c.c. of 17 per cent. sodium hydroxide solution for one to two hours, 100 c.c. of water are then added, the mixture is passed through a porcelain filter, and 100 c.c. of the clear filtrate transferred to an Erlenmeyer flask. The residue on the filter is washed with water, digested with 5 per cent. acetic acid, washed till neutral, and then treated twice more with sodium hydroxide solution as before, 100 c.c. from each filtrate being added to the original amount in the flask. The solution is neutralised with concentrated nitric acid and then heated on a water-bath for three hours with 125 c.c. of 20 per cent. nitric acid and 500 c.c. of water. The residue is collected on a tared filter, washed with warm water till neutral, dried at 100° C. till constant, and weighed. This method is preferable to the determination of the copper number, which gives only empirical results. Hemi-celluloses are identified by converting them into sugars by heating for five hours with 4 per cent. hydrochloric acid. For the estimation of xylose, derived from xylan by the furfural distillation, an improved apparatus has been devised. It comprises a 300 c.c. round-bottomed flask with a long neck, into which the still-head is closely fitted by means of a ground-glass joint and maintained in position by a spring-clip. The still-head is bulb-shaped and terminates above in a funnel and stopcock. A lateral tube is fused into the bulb with its end projecting slightly into the interior and bent upwards, and the other end of the tube has a bead at a right angle passing through a rubber bung into a Liebig condenser. A graduated measuring flask receives the distillate. The heating bath consists of calcium chloride solution with a surface layer of vaseline or paraffin to check evaporation. The furfural-yielding constituents of cellulose exist partly as xylan and partly as a more firmly combined complex compound. The presence of mannan is indicated by the formation of mannose on hydrolysis with weak hydrochloric acid; mannan is more easily separated than xylan.

W. J. W.

Estimation of Hydrochloric Acid and Neutral Chlorides in Leather.

A. W. Thomas and A. Frieden. (*J. Ind. Eng. Chem.*, 1920, 12, 1186-1188).—*Total Chloride*: One gram. of the leather is mixed with 200 c.c. of 0.1M sodium dihydrogen phosphate solution and heated for two hours in a boiling water-bath. The mixture is then diluted to 250 c.c., and filtered. Two hundred c.c. of the filtrate are acidified with nitric acid, silver nitrate solution is added, and, after the addition of 20 c.c. of concentrated nitric acid, the mixture is evaporated in the dark to about 50 c.c. in order to oxidise and dissolve organic matter which is precipitated with the silver chloride; the solution is diluted and the silver chloride collected, dried, and weighed. *Neutral Chloride*: Another portion of 1 gram. of the sample is digested at 75° C. with 200 c.c. of 95 per cent. alcohol for two hours, cooled, diluted with alcohol to 250 c.c., and filtered; 200 c.c. of the filtrate are rendered alkaline with sodium hydroxide, evaporated to expel alcohol, the residual solution is acidified with nitric acid, treated with silver nitrate and nitric acid, and the estimation finished as described above. The amount of silver chloride obtained in this estimation is equivalent to the neutral chlorides present, and the difference between the amount of neutral chloride and total chloride is a measure of the acid chloride present in the leather. W. P. S.

Qualitative Detection of certain Naphthalene Disulphonic Acids.

J. A. Ambler. (*J. Ind. Eng. Chem.*, 1920, 12, 1194).—A portion of the acetone-insoluble dry β -naphthylamine salts of the acids is boiled with 5 c.c. of 80 per cent. alcohol, the solution, if not clear, is filtered and allowed to cool; the β -naphthylamine salt of 2-7-disulphonic acid crystallises out. Another portion of the dry salts is boiled with a small quantity of water and β -naphthylamine removed by titrating the solution with $\frac{N}{1}$ sodium hydroxide solution, phenolphthalein being used as indicator. The separated amine is filtered off, the filtrate evaporated to dryness, and the residue is dissolved in a volume of water equal to that of the sodium hydroxide solution used for the neutralisation. The mixture is then treated with its own volume of concentrated sulphuric acid, cooled, and placed aside for several hours. The sodium salt of the 1-6-disulphonic acid crystallises out slowly, whilst the 2-7-acid remains in solution. W. P. S.

Analysis of Commercial Casein. **R. H. Shaw.** (*J. Ind. Eng. Chem.*, 1920, 12, 1168-1170).—The following procedure is recommended for the estimation of moisture, ash, sugars, fat, phosphorus, and calcium in casein: *Moisture*.—A weighed portion of the sample is heated at 100° C. under reduced pressure until constant in weight. *Ash*.—Three grms. of the sample are moistened in a silica basin with 5 c.c. of calcium acetate solution (yielding 0.15 gram. CaO per 5 c.c.), the mixture is dried, charred carefully, and then heated at a low red heat until a white ash is obtained; the amount of CaO introduced as calcium acetate is deducted from the weight of the ash. *Fat*.—A slight modification of the Rose-Gottlieb method is used: One gram. of the casein is shaken for fifteen minutes with 10 c.c. of water; 2 c.c. of ammonia are then added, followed after ten minutes by 10 c.c. of 95 per cent. alcohol, and the process is completed in the usual way. *Sugar*.—Ten grms. of casein are shaken for

four hours with 250 c.c. of 50 per cent. alcohol, the mixture allowed to stand for several hours, the liquid portion then submitted to centrifugal action, and the clear solution obtained is used for the estimation. *Phosphorus and Calcium*.—Phosphorus is best estimated by the official (American) method for the estimation of total phosphorus in fertilisers; all the calcium is found in the ash and its estimation presents no difficulty.

W. P. S.

Standardisation of the Borax Solubility Test for Commercial Caseins.

H. F. Zoller. (*J. Ind. Eng. Chem.*, 1920, 12, 1171-1173.)—Fifteen grms. of the casein, ground to pass a 40-mesh sieve, are mixed with 100 c.c. of 0.2M borax solution at 30° C. (76.32 grms. of crystallised borax per litre) and the mixture is stirred every five minutes for half an hour. The character of the solution is observed during this period; the relative various caseins gave the following results:

Commercial sulphuric acid casein (made at 120° F.; ash, 5.04 per cent.): After 15 minutes, stiff, clear, gel undissolved; after 30 minutes, stiff, clear, gel not completely dissolved. *Natural sour casein* (made at low temperature; ash, 3.69 per cent.): After 15 minutes, smooth, mucilaginous, largely dissolved; after 30 minutes, smooth, mucilaginous solution. *Kahlbaum's casein* (low temperature): After 15 minutes, smooth, clear, mucilaginous solution; after 30 minutes, smooth, clear, mucilaginous solution. *Natural sour casein* (high fat): After 15 minutes, gelatinous, undissolved, milky white; after 30 minutes, mostly dissolved, milky, poor cohesion. *Grain curd hydrochloric acid casein* (made at 95° F.; ash, 2.55 per cent.): After 15 minutes, clear, smooth, mucilaginous solution; after 30 minutes, clear, smooth, mucilaginous solution. *Natural sour buttermilk casein* (made at 120° F.; fat, 4.0 per cent.): After 15 minutes, stiff gel, undissolved, slightly milky; after 30 minutes, gelatinous, mostly undissolved, poor cohesion.

The viscosity curve of casein in borax solutions shows that the maximum viscosity is obtained at a hydrogen ion concentration of P_H 8.15, whilst at P_H 9 the viscosity is less but constant, owing to the buffer effect of the borax in this region. The differences in the physical structure of caseins have greater influence on their viscosity than have the normal contaminating substances present in commercial caseins.

W. P. S.

Micro-sections Cut from Vulcanised Rubber Articles. **H. A. Depew and I. R. Ruby.** (*J. Ind. Eng. Chem.*, 1920, 12, 1156-1159.)—Microscopical examination of sections cut from rubber articles affords useful information regarding the quality of the substance; flocculated pigment (which accounts for the heating up of tyre treads heavily compounded with carbon black) is readily detected, as is also the presence of reclaimed rubber, fibre, etc. In some cases the source of the pigment may be ascertained by microscopical examination; for instance, chemical analysis shows no difference between *blanc fixe* and ground baryta, but they are readily distinguished under the microscope. The sections are best cut after the rubber has been fixed to a slide with glycerol-water (9:1) solution and then frozen, first by expansion of compressed carbon dioxide, and then with liquid air; the freezing must be just sufficient to destroy the elasticity of the rubber, and yet not make it too hard.

W. P. S.

Relation of Soil Solution to the Soil Extract. D. R. Hoagland, J. C. Martin, and G. R. Stewart. (*J. Agric. Research*, 1920, 20, 381-395.)—Soil extract contains the soluble matter present in the soil solution in addition to other substances derived from adsorbed or readily soluble soil components. The amounts of the latter substances depend upon the concentration and composition of the soil solution, since the soil constituents appear to be less soluble when large amounts of soluble substances are present. The detailed investigations of the authors indicate that soil extracts should be made with a small proportion of water, such as 1 part of water to 1 of soil where practicable, and for a short period only. It is probable that the ratios between the soil solution and the concentrated soil extracts are similar for the majority of the important elements present, and analyses and freezing-point determinations of suitable soil extracts enable the approximate concentration and composition of soil solutions to be estimated. T. J. W.

A New Indicator. R. W. Kinhead. (*Chem. News*, 1921, 122, 4-5.)—The new compound was prepared by gradually adding one equivalent of ethyl nitrate to one equivalent of phenyl magnesium bromide cooled in ice, when in a short time a vigorous reaction took place. On the following day ice was added, the precipitate allowed to settle, and the ethereal layer evaporated. Water and solid sodium carbonate were then added and the solution extracted with chloroform, the blue sodium salt remaining in the aqueous layer. Since the colouring matter is unstable, except in very dilute aqueous solution, the blue solution was acidified and extracted with ether, the ethereal solution being suitable for use as an indicator. The substance has not been prepared in a pure condition. The indicator gives a sharp end point, changing from red to blue on the addition of 0.05 c.c. of N_{10} sodium hydroxide, but is useless in the presence of carbon dioxide. Sulphur dioxide reduces the substance both in acid and alkaline solution, but hydrogen peroxide does not appear to affect it. T. J. W.

INORGANIC ANALYSIS

Determination of the Conductivity of Electrolytes. A. H. W. Aten. (*Chem. Weekblad*, 1921, 18, 51-52.)—The conductivity of electrolytes has been determined by a method the essential feature of which is the employment of a differential galvanometer and a variable resistance, and in which certain disadvantages of the Kohlrausch method are overcome. Errors arising from polarisation and the heating effect of the current may be reduced by increasing the surface of the electrodes and the volume of the electrolyte, and become smaller with higher resistance. Comparison of this method with Kohlrausch's method indicates that with a resistance not below 100 ohms an accuracy of 0.3 per cent. is obtainable. The method has a special application in certain technical analyses, such as estimation of the ash in sugar and of chlorides in drinking water, the determination of carbon dioxide in chimney gases, and the selection of the end point in the evaporation of condensed milk. W. J. W.

Catalysis in Permanganate Titrations. P. H. Segnitz. (*J. Ind. Eng. Chem.*, 1920, 12, 1196-1197.)—Results of experiments indicate that the addition of

manganous sulphate to a solution to be titrated with permanganate solution accelerates the reaction velocity, so that the time of the peroxide-permanganate titration can be reduced from about forty-five minutes to eight seconds, and that of the cold oxalate-permanganate titration from over an hour to six minutes. The use of the catalyst does not interfere with the end point of the titration. W. P. S.

Preparation of Anhydrous Stannic Chloride. C. van Loon. (*Chem. Weekblad*, 1920, 17, 664.)—In the preparation of anhydrous stannic chloride (*Chem. Weekblad*, 1920, 17, 610) in small amounts, quantitative results may be obtained by using a round-bottomed flask instead of the wide tube employed by Lorenz (*Zeitsch. anorg. Chem.*, 1895, 10, 44). W. J. W.

Solubility of Metals in Acids containing Formaldehyde. R. C. Griffin. (*J. Ind. Eng. Chem.*, 1920, 12, 1158-1160.)—The presence of 1 per cent. of formaldehyde in dilute sulphuric acid (sp. gr. 1.075) and in dilute hydrochloric acid (1 : 1) considerably decreases the solvent action of these acids on wrought iron, cast iron, and steel, the corrosion loss being diminished 50 to 95 per cent. The effect in the case of other metals (manganese, bronze, tin, brass, nickel, etc.) is much less, and it is also less with 10 per cent. nitric acid, possibly on account of secondary reactions. W. P. S.

Gasometric Estimation of Nitrogen. R. L. Stehle. (*J. Biol. Chem.*, 1920, 45, 223-228.)—The sample is digested with a small quantity of sulphuric acid and a crystal of copper sulphate, and on cooling the solution is diluted to 100 c.c. Ten c.c. are run into a nitrometer containing mercury, and connected at the lower end by a long indiarubber tube to an adjustable mercury reservoir, and dissolved air is liberated by reducing the pressure and expelled from the apparatus. A strong solution of alkali is run in to neutralise the acid, and 2 c.c. of a solution prepared by mixing equal volumes of 28 per cent. sodium hydroxide and a solution containing 12.5 per cent. of sodium bromide and 12.5 per cent. of bromine, the mixture being diluted with three volumes of water, are added. The pressure in the reaction chamber is reduced, and the nitrogen is completely evolved by shaking the nitrometer for a minute. Since a small amount of oxygen is liable to be set free, this gas is absorbed by running in 1 c.c. of sodium pyrogallate solution. The mercury is adjusted until the volume of nitrogen is exactly 1 c.c., and the weight of nitrogen present is calculated from the observed temperature and pressure. The method yields excellent results with substances of known nitrogen content, but a maximum error of about 8 per cent. is shown by comparison of this method with the usual Kjeldahl estimation when estimating the non-protein nitrogen in blood. T. J. W.

Properties of Pure Hydrogen Peroxide. O. Maass and W. H. Hatcher. (*J. Amer. Chem. Soc.*, 1920, 42, 2548-2569.)—Pure (99.9 per cent.) hydrogen peroxide was prepared from an impure 3 per cent. solution by concentration in a sulphuric acid vacuum concentrator (*ANALYST*, 1920, 68), distillation, and

fractional crystallisation. It is almost insoluble in anhydrous ether, dissolves various anhydrous salts readily, and, on heating, is decomposed in contact with glass. Most metals cause decomposition of the pure substance, but not explosively. Pure aqueous solutions in a suitable container keep indefinitely, but are decomposed by foreign substances gaining access to the solution. Strong solutions are more stable than dilute, as the former more readily destroy impurities, and thus prevent further decomposition. Investigation of so-called "anti-catalysers" showed that in all cases these substances acted by destroying others, which caused decomposition, and that spontaneous decomposition of pure hydrogen peroxide did not occur (*cf.* ANALYST, 1896, 20, 38).

T. J. W.

Micro-chemical Identification of Gaseous Ammonia as Hexamethylenetetramine Picrate. C. Kollo and V. Teodossiu. (*Bull. Soc. Chim. România*, 1920, 11, 100-102.)—When a drop of a solution of formaldehyde (commercial formalin), together with a drop of a 1 per cent. solution of picric acid, is exposed to ammonia, hexamethylenetetramine is produced, and combines immediately with the picric acid to form characteristic crystals of the picrate. The test is not sufficiently delicate when carried out in this way, as ammonia picrate is formed in the presence of much ammonia, whilst in the presence of small amounts of ammonia the formation of hexamethylenetetramine picrate only takes place on rubbing, and the crystals break up and lose their characteristic form. A highly sensitive reagent is prepared by saturating a solution of formaldehyde in the cold with picric acid, and then saturating this with hexamethylenetetramine picrate. The ammonia liberated from 1 mgrm. of ammonium chloride may be detected by exposing one drop of the reagent for five to six minutes to the action of the gas under examination.

R. G. P.

New Method for the Separation and Estimation of Iron and Manganese. C. Kollo. (*Bull. Soc. Chim. România*, 1920, 11, 89-95.)—The method of separation is based on the precipitation of ferric hydroxide when hexamethylenetetramine is added to a slightly acid solution of an iron salt, the manganese forming soluble double salts of the type $Mn(CH_2)_6N_4 \cdot 2H_2SO_4$, manganese oxide only being precipitated in the presence of hexamethylenetetramine from a neutral solution on boiling. To carry out the separation the solution of ferric and manganese salts is slightly acidified, and a 10 per cent. solution of hexamethylenetetramine is added until the supernatant liquid does not change the colour of Congo-red paper, after which the liquid is warmed gently on a water-bath to cause the ferric hydroxide to flocculate. After removal of the manganese salts by washing on a filter, the iron is determined gravimetrically as usual, or by dissolving the ferric hydroxide in hydrochloric acid, adding excess of potassium iodide and titrating the liberated iodine with standard thiosulphate solution. The manganese in the filtrate may be determined as manganese sulphate after evaporation and ignition, or by Volhard's method after the destruction of organic matter by evaporation with sulphuric acid. The following results were obtained by these methods:

Salts.	Weight taken.	Weight found—	
		Gravimetrically.	Volumetrically.
	Grm.	Grm.	Grm.
$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$	0.1298	0.1300	0.1284
$\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$	0.1406	0.1374	0.1412
$\text{FeCl}_3 \cdot 12\text{H}_2\text{O}$	0.4950	0.5139	0.5101
$\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$	0.2200	0.2300	0.2285

R. G. P.

The Use of Phenolphthalein and Diphenylamine in the Estimation of Manganese by the Persulphate Method. D. H. Wester. (*Rec. Trav. Chim. Pays-Bas*, 1920, **39**, 600-602.)—The substitution of an alkaline phenolphthalein solution for permanganate (Tillmans and Mildner, *J. Gasbeleucht. u. Wasserversorg.*, 1914) in the estimation of manganese gives inaccurate results, probably owing to variation in the alcoholic strength of the phenolphthalein; in extreme cases this may be 2 per cent. to 25 per cent. With constant alkaline concentration, the violet coloration is affected more by the content of water than of phenolphthalein in the solutions, and increase of the phenolphthalein does not, therefore, give a proportionately deeper intensity of colour. No better results are obtained with diphenylamine. The blue coloration disappears rapidly, and there is no relation between its intensity and the amount of manganese.

W. J. W.

Micro-chemical Identification of Potassium and Sodium in Presence of Magnesium. E. Ludwig and H. Spirescu. (*Bull. Soc. Chim. Roumanie*, 1920, **11**, 78-82.)—Calcium, strontium, and barium are first removed by precipitation with ammonium carbonate or with ammonium oxalate and sulphuric acid, the filtrate evaporated, and the residue ignited until free from ammonium salts and used for the detection of potassium and sodium. The complex nitrite $\text{K}_2\text{CuPb}(\text{NO}_2)_6$ is employed as a reagent for potassium, the test for which is carried out by placing a drop of sodium nitrite solution (20 grms. in 75 c.c. of water), and a drop of copper and lead acetate solution (copper acetate 9.1 grms., lead acetate 16.2 grms., acetic acid 2 c.c., water 75 c.c.) on the slide and adding a fragment of the residue under examination. In the presence of potassium the complex nitrite is precipitated in the form of cubic black crystals, even in the presence of large amounts of magnesium, whilst the reaction is more delicate than that with platinic chloride. For the detection of sodium by means of potassium pyroantimoniate, the soluble magnesium salts (which interfere with the reaction) are converted into insoluble basic magnesium carbonate by adding a drop of concentrated potassium carbonate or bicarbonate to a fragment of the residue and evaporating the mixture to dryness on the slide. On adding a drop of potassium pyroantimoniate solution, a characteristic precipitate of sodium hydrogen antimoniate ($\text{Na}_2\text{H}_2\text{Sb}_2\text{O}_7$) is formed.

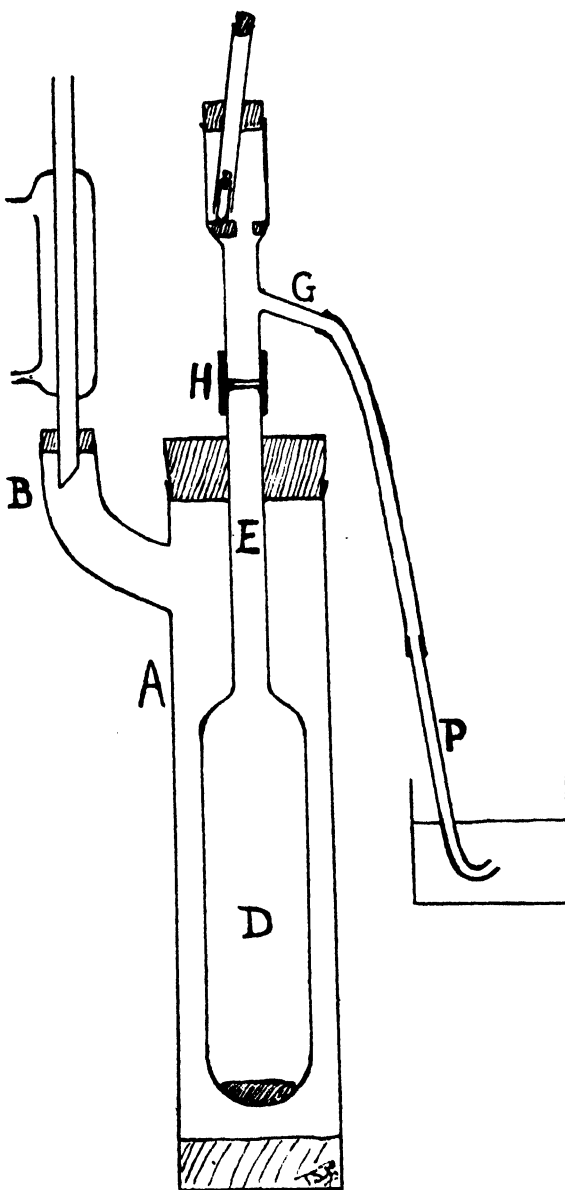
R. G. P.

APPARATUS, ETC.

Modification of Victor Meyer's Vapour Density Apparatus. T. S. Patterson.

(*Chem. News*, 1920, 121, 307.)—The apparatus described is stated to yield very accurate results. The outer glass jacket is replaced by a cylinder of copper, having the lower end closed by brazing, and provided near the top with a short side tube, into which is fitted a small condenser. To facilitate the fitting of the large jacket cork the inner tube is cut into two pieces a short distance below its side tube, the upper and lower portions being again joined by a piece of stout indiarubber tubing. The small tube containing the substance under examination is allowed to fall down the inner tube by means of an ingenious device previously described by the author (*ANALYST*, 1908, 33, 207), fracture being prevented by a small quantity of mercury at the bottom of the inner tube.

T. J. W.

**Apparatus for Generating Hydrogen Sulphide. V. B. Connell.**

(*Pharm. J.*, 1921, 106, 17.)—The apparatus comprises two wide-mouthed bottles, having well-fitting rubber bungs with two perforations, one bottle being inverted above the other. The lower bottle contains a layer of

asbestos and ferrous sulphide lumps, and is half filled with a mixture of 1 part of sulphuric acid and 3 parts of water. A U-tube passes through the bung of the upper vessel with its long limb reaching nearly to the top, and another U-tube passes just through the bung of the lower vessel and is connected externally, by means of

tubing and pinchcock, to a delivery tube. Communication between the two bottles is established by a tube reaching to the bottom of the lower one and just extending through the bung of the upper one. In operation, when the pinchcock is closed, the pressure of the gas generated in the lower bottle forces the liquid into the upper one and expels the air through its U-tube. When the pinchcock is opened the liquid falls again and thus expels the gas through the delivery tube, after which evolution of gas recommences.

W. J. W.

Michell's Cup and Ball Viscosimeter.* T. C. Thomsen. (*Report of Lubrication Inquiry Committee, Dept. Scientific and Ind. Research, 1920, 15-16, 103-110.*)—This apparatus consists of a steel cup and a loose steel ball about $\frac{3}{4}$ -inch in diameter. A few drops of oil are introduced into the cup, and the ball inserted and pressed firmly into position, three pegs standing out from the surface a few thousandths of an inch separating it from the wall of the cup. After removal of the surplus oil the apparatus is inverted, the ball being meanwhile kept in position with the finger, and a note is taken of the time before the ball falls out of the cup. The results obtained with this instrument are calculated into seconds on Redwood's viscosimeter by means of the formula— $kt\bar{d} = \eta$, where k is the constant; t , time of outflow; $\bar{d}$, the sp. gr. at the time of outflow; and η , the absolute viscosity. The readings are greatly influenced by variations in the temperature, especially in the case of oils of high viscosity, and are sometimes as much as 15 per cent. above the true viscosity. By immersing the instrument in oil, however, and keeping the temperature at 70° F. for about half an hour, results may be obtained agreeing within 1 per cent. of the true viscosity, as measured by standard instruments for oils with absolute viscosity ranging from 0.8 to 5.

Deeley's Oil-Testing Apparatus.* (*Report of Lubrication Inquiry Committee, Dept. Scientific and Ind. Research, 1920, 15, 101-103.*)—This machine consists essentially of a horizontal disc, about 3 inches in diameter, resting in a circular pan secured to the end of a vertical spindle, which can be rotated by hand or mechanically. Upon the horizontal disc rest the lower flat surfaces of three vertical pegs secured to an upper disc, to which is fixed two or more uprights, and the two discs are pressed together by means of weights placed upon the upper one. Above the weights and concentric with the power spindle is a vertical shaft, with which is connected a coil spring, an indicating finger, a free train of wheels, and a cross arm forming a universal joint which engages with the uprights secured to the loaded disc. The extent to which the spring is coiled affords a measure of the friction between the disc and the pegs, and this is shown by the indicating finger upon a scale. Oscillation of the finger through rapid changes of friction is prevented by the free train of wheels. The apparatus is suitable for the measurement of static and low-speed kinetic friction between flat surfaces of metals when dry or fully lubricated and under various loads. Results obtained with different oils have proved that vegetable oils have smaller frictional resistance than mineral oils, which is in accordance with the fact that has long been known from practical experience that the lubricating value of the former is greater.

* By permission of H.M. Stationery Office.

REVIEWS.

ORGANIC MEDICINAL CHEMICALS. M. BARROWCLIFF, M.B.E., F.I.C., AND FRANCIS H. CARR, C.B.E., F.I.C. INDUSTRIAL CHEMISTRY SERIES. Edited by S. RIDEAL, D.Sc., F.I.C. Pp. 331. London: Baillière, Tindall and Cox. Price 15s. net.

A few months ago, the present writer had the pleasant task of reviewing the "Extra Pharmacopœia" of Martindale and Westcott for the ANALYST; his present duty is similar in some respects, in others very different. Messrs. Barrowcliff and Carr treat the subject chiefly as a branch of manufacturing chemistry, and our technical literature receives a welcome addition.

General Preface and Author's Preface are followed by a short Introduction, which, brief as it is, shows clearly how the development of Organic Chemistry and the treatment of disease go forward hand in hand. The work itself is divided into eleven sections, dealing respectively with (I.) Narcotic and General Anæsthetics; (II.) Naturally occurring Alkaloids and their Derivatives; (III.) Natural and Synthetic Local Anæsthetics; (IV.) Antipyretics and Analgesics; (V.) Organic Antiseptics and Disinfectants; (VI.) Purgatives; (VII.) Vaso-Constrictors and Vaso-Dilators; (VIII.) Diuretics and Uric Acid Solvents; (IX.) Organo-Metallic Compounds; (X.) The Digitalis Group, Skin Irritants, Glucosides, and Neutral Principles; (XI.) Other Substances of Interest.

It will be noticed that the classification of subject-matter is more in accordance with the uses for which the materials are employed than with their chemical structure, and for most purposes this is the more convenient course to follow. The authors leave no doubt, however, that the work deals with a branch of Industrial Chemistry of which they possess a very expert knowledge, and directions are given for the preparation of most of the compounds described in the book. Reference is made to the literature of the subject, whether it has appeared in purely scientific journals or in patent specifications, though much of the information is evidently derived from first-hand knowledge, and is especially useful in a book of this description.

As an example of the method followed by the authors one may take the case of Beta-eucaine (Benzoylvinyl-diacetonalkamine). A scheme of synthesis is placed first, showing that the necessary organic materials are acetone, acetal, and benzoyl chloride. Then follows the preparation of diacetonamine from acetone and ammonia, Everest's method being employed; the conversion of diacetonamine acid oxalate into vinyl-diacetonamine oxalate according to the patent of King, Mason, and Schryver comes next, then the reduction to the alkamine (Robinson's Communication to the Royal Society Committee), and finally the benzoylation is described.

The book will be very useful, for it is well written, well indexed, and illustrated by twenty-five drawings or diagrams of plant.

J. T. HEWITT.

THE YEAR-BOOK OF PHARMACY. Pp. 594. London: J. and A. Churchill. 1920.
Price 12s. 6d. net.

To review a work which has been published annually for so many years might seem a work of supererogation, were it not for the fact revealed by a glance at the list of members of the British Pharmaceutical Conference, of which the Year-Book is the organ, that comparatively few public analysts give their support to this body. Public analysts are appointed under the Sale of Food and Drugs Act, and it is hard to see how they can do without the annual volume, which epitomises the year's work on Pharmacy, Materia Medica, and Chemistry of Drugs; at least half this book contains matter in which the public analyst will find a direct interest, while the remainder will help him to understand how the pharmacist carries on his business, a knowledge which may have a real importance when the question is considered of advising as to what action should be taken as the result of analysis of a sample.

The plan of the book is somewhat unusual, in that the Abstracts of papers contributed to scientific journals from July 1, 1919, to June 30, 1920, occupy the first portion; the first section deals with chemistry, divided into convenient sub-sections, and this is followed by Materia Medica and Pharmacy, each similarly split up in sub-sections, and a very useful research list; as is only to be expected with the advance of knowledge, the last is a little out of date, more than one of the subjects suggested for investigation having been elucidated by work which is later than the middle of 1920.

The remainder of the book consists of the Transactions of the British Pharmaceutical Conference at Liverpool in July, a list of members, and a good and efficient index.

H. DROOP RICHMOND.

PRACTICAL PHYSIOLOGICAL CHEMISTRY. By SYDNEY W. COLE, M.A. Sixth Edition.
Cambridge: W. Heffer and Sons, Ltd. Price 15s. net.

As Professor Hopkins points out in his introduction to this volume, there are periods in the growth of any branch of knowledge when the development of technique becomes the most pressing of needs, and its success the best measure of progress. Physiological chemistry has in recent years been passing through such a stage, and its methods have been very largely multiplied and improved. Hence the necessity of a sixth edition of this deservedly popular book only a year after the publication of the fifth, which itself included a very considerable amount of new material. In the main the book represents the course in practical physiological chemistry for medical students at Cambridge, and contains not only all the common biochemical methods required by the medical student, but also the more important of those used in medical diagnosis and biological researches. The methods described have been thoroughly tested in the laboratory; older methods have been modified in detail as a result of experience of their use in class, and not a few of the processes described are original—indeed, the book has been used as a medium for the publication of much patient research. The most important additions to the fifth edition were chapters on the properties of solutions, in which particular attention is paid to hydrogen ion concentration and properties of colloids; on the preparation and properties of amino acids;

on the preparation and hydrolysis of nucleic acid; and on the action of oxidase systems and new quantitative methods related to enzyme action and blood, urinary and gastric analyses.

The chapters on solution and on amino acids afford perhaps the best examples of the way in which Mr. Cole tests all processes described. The preparations of the amino acids are given with such a wealth of essential detail that even a beginner could scarcely fail to carry them out successfully.

A welcome feature of the present edition is the introduction of several methods of micro-analysis. It may perhaps be open to doubt how far such methods can be introduced with advantage into an elementary course, but there can be no doubt that the more advanced student should have practice in well tried micro-methods and gain a practical knowledge of the precautions and care necessary to attain reliable results and of their limitations, since such methods are often the only ones available in the study of the biochemistry of the living subject without terminating the life of the animal or injuring the human subject.

Among the more important of the methods described we may mention a modification of McLean's method for the determination of blood sugar devised by the author, and what he states has been used for the past year in his classes with very concordant results. It is offered as a substitute for Bang's micro-method, which, though it may be reliable in the hands of a practical analyst, is probably beyond the powers of the average student.

The hypobromite method for estimating urea, omitted in the former edition, has been again included, though the results are unreliable, on the ground that it is sufficiently accurate for most clinical work and demands very little equipment. The use of the enzyme urease, which has rendered obsolete a large number of methods for the estimation of urea in urine is illustrated by Van Slyke's micro-method. The urease method on the ordinary scale is not mentioned, though it was described in this country before Van Slyke's micro-method was published.

We hope that the author in his next edition will see his way to extend the chapter on fats, which by comparison with some of the other subjects seems to us to be dealt with rather inadequately. Though the aim of the book is essentially practical there is judicious reference to theory, and, without undue consumption of space, to the significance of results. In our opinion the book is a contribution of substantial importance in connection with the teaching of biochemistry. It should be of great use to all students of this subject, whether their ultimate aim be medicine, or agriculture, or the career of a professional analyst. It should also be a welcome addition to the library of all who may have occasion to seek for accurate and full descriptions of biochemical methods outside his own special field.

S. B. SCHRYVER.

A TEXTBOOK OF ORGANIC CHEMISTRY. By A. F. HOLLEMAN. Edited by A. Jamieson Walker. London: Chapman and Hall. 1920. Price 18s. 6d. net.

This textbook is too well known and too widely used to be in need of much further recommendation.

In the present new edition numerous small alterations have been made, the out-

standing one being the additional space devoted to the applications of physico-chemical methods in organic chemistry. The importance of such properties as refraction, absorption, and viscosity in organic chemical research is, as the author remarks, steadily increasing.

One of the most valuable features of the book is the attention which is given to considerations of the structure of most of the representative compounds; an excellent example of the thoroughness with which this important subject is dealt is to be seen on pp. 152-158, where the structure of unsaturated compounds is passed under review.

The chapter on the constitution of benzene has been enlarged, and contains a short account of Willstätter's attempted synthesis of cyclo-hexatriene; the fact is emphasised that the compound he obtained resembled benzene in all respects, whereas cyclo-octatriene prepared in a similar manner by Willstätter is a highly unsaturated compound. The conclusion is drawn from this and other considerations that Thiele's modification of Kekulé's formula is the best available representation of the structure of the benzene molecule.

At the end of the section dealing with optically active compounds a very brief account has been introduced of Werner's theory of the stereoisomerism of complex derivatives of certain metals.

The difficult subject of the Walden inversion has been given a little more space so as to include a short account of Stark's hypothesis, according to which the union of carbon atoms is due to "valency electrons."

The arrangement of the material is good, the style is flowing and easy, the print is clear and pleasing to the eye, and a comprehensive index is provided. Altogether author, editor, and publishers, have combined to produce an excellent and eminently readable volume.

JOSEPH KENYON.

THE VOLATILE OILS. By GILDEMEISTER and HOFFMANN. Second Edition by E. GILDEMEISTER, translated by EDWARD KREMERS. Vol. II. Pp. xx + 686. London: Longmans, Green and Co. Price 32s. net.

This volume of the well-known German work on Essential Oils deals, in a series of monographs, with the first half of the essential oils grouped according to Engler's "*Syllabus der Pflanzenfamilien*." Analytical methods are only dealt with incidentally as occasion requires. It will be well to remember the position of this volume in the series. Volume I., published in 1910, deals with the history of certain essential oils, the constituents of essential oils, synthetic perfumes, and the analysis of essential oils, to which about a hundred pages are devoted. The present volume was written in 1913-14, printed in 1916, and published at the end of 1920. Volume III., which completes the study of the individual essential oils, appeared in German during the period of the war.

The work is one of such a high degree of merit, so free from mistakes, so thorough and exhaustive that no commendation can be too high for it, the only hostile criticism possible being that the present volume, published in 1920, is practically seven years out of date, so that, although accurate and complete in 1914, it is now so full of omissions and of statements which the last seven years' work

has shown to be inaccurate or insufficient, that as a work of reference it must be taken with considerable care and reserve. This, however, refers to the study of the individual essential oils, rather than to analytical methods, and therefore need not be here discussed further.

From a market point of view, no essential oil is of such importance in regard to its analysis as oil of turpentine. We should therefore expect the most exhaustive treatment of the matter here. The iodine absorption of this oil has received a considerable amount of attention, and the determination of this value on the highest fraction of the oil shows so large a difference between normal oil of turpentine and so-called "stump" turpentine, due to the accumulation of practically saturated bodies in the last fraction in the case of stump turpentine, that it may almost be regarded as the decisive feature of the analysis. This is not referred to, although the iodine value of the oils themselves is mentioned. The information under Russian turpentine oil will not give the slightest assistance to the analyst who has to deal with market samples of this oil. Yet very full information on the characters and analysis of this oil was published some ten years ago on samples brought from Russia by Professor Schindelmeyer, and on market samples, in comparison.

It is a matter of regret that the acetylation process is still described as it was in the first volume in 1910, where equal volumes of the oil and of acetic anhydride are directed to be used. In dealing with oils containing 80 to 90 per cent. of acetylisable constituents, there is no doubt that this results in too low values, and 10 volumes of the oil and 15 volumes of acetic anhydride must be used. Still higher results are obtained if the amount of acetic anhydride be further increased, but as this is possibly due to secondary reactions, 10 and 15 volumes have been universally agreed to in this country, and, we believe, in America.

Considering the indefinite nature of most organic processes of analysis, together with the personal error, it seems rather absurd to give the figure 0.19974314 as the figure to multiply the "amount of iodine consumed" to obtain the corresponding amount of methyl salicylate. We should have preferred the simpler 0.2!

On page 481 it is stated that the percentage of alcohol (linalol) in cayenne linalol oil can be determined by acetylating for seven hours in xylene solution. No acetylation of linalol is accurate, although in xylene dilution it becomes more nearly correct, and it should be mentioned that this figure is merely an approximation.

Otto of rose is, of course, a most difficult oil upon which to express an opinion, but the upper limit of specific gravity for Bulgarian oil—namely, 0.862 at $\frac{20}{15}$ —indicates, almost with certainty, that the oil is adulterated. The presence of gurjun balsam oil as an adulterant in otto of rose was first proved by Schimmel and Co., after having been indicated by the reviewer, and confirmed by Umney. This body appears to have found its way into otto of rose through being used as an adulterant of palmarosa oil, which is commonly used as the source of geraniol for adulteration purposes. It can be detected with certainty by a well-marked colour reaction, but this is not referred to, although no other means of detection, except by tedious research on large quantities of the oil, is available. There is a good deal in the subject-matter of otto of rose with which the reviewer disagrees, but it does not come within the scope of this review.

Apart from a few matters such as those indicated and the fact of the book being six or seven years out of date, one can only say that this is a work of very high merit, which will, as a matter of course, take its place on the bookshelf of everyone interested in essential oils.

ERNEST J. PARRY.



FIRST INTERNATIONAL CHEMICAL CONFERENCE.

THE first International Chemical Conference was held in Rome from June 22 to June 24, 1920, under the presidency of Professor Charles Moureu. The Council of the International Union of Pure and Applied Chemistry, composed of representatives of Belgium, France, Great Britain (Sir William Pope and Mr. Hay), Italy, and the United States, with Mr. J. Gerard as General Secretary, admitted Canada, Czechoslovakia, Denmark, Greece, Holland, Poland, and Spain to the Union, and the General Assembly was then opened.

The Union is administered by a Council composed of delegates from each of the adherent countries, its executive power being entrusted to a Bureau which works through a permanent International Office of Chemistry with a general secretary. A joint Session of the Council, Permanent Committee, Consulting Committee, and General Assembly is to be held each year under the title of the "International Chemical Conference."

The Conference decided that the Council of the Union should organise a bureau of standards, the three sections of which (chemical standards, pure products for research, and technological products) should be entrusted to Belgium, Great Britain, and France, respectively, with the United States forming an allied branch. A sub-committee was appointed to deal with the subject of thermo-chemical standards. Problems relating to patents and the question of the establishment of an international patent bureau were placed in the hands of an Italian Committee.

It was agreed to form an International Committee on atomic weights, upon which Sir Edward Thorpe and Messrs. Clarke and Urbain should be invited to serve. The following motions were carried: (1) That atomic weights be revised decennially. (2) That Dalton's proposal, accepted by Avogadro and Cannizzaro, of basing the atomic weights upon hydrogen, $H = 1$, be again adopted as the basis of the system.

In accordance with a motion of the English Chemical Society, the Union was requested to ask the Chemical Associations and journals of the countries within the Union to republish the lists of physico-chemical symbols adopted by the old International Association of Chemical Societies at the third Session of their Council in Brussels in 1913.

It was decided to accept the invitation of the Polish Government to hold the next Conference in 1921 at Warsaw.



INTERNATIONAL PHYSICO-CHEMICAL SYMBOLS.

SYMBOLS suggested, but not definitely recommended, and alternative symbols suggested for use where there is a possibility of confusion are placed in brackets.

1. *General Physics and Mathematics*.—Acceleration due to gravity, g ; Angström unit (10^{-10} metre), $\text{\AA}$; Area, q ; Base of natural logarithms, e ; Co-ordinates, variables, x, y, z ; Critical quantities: pressure, p_c , volume, v_c , temperature (Centigrade), t_c , temperature (absolute), T_c , density, d_c ; Density (mass per unit vol.), d ; Diameter, (d) ; Differential sign, total, d ; Differential sign (partial), ∂ ; Fluidity, (ϕ) ; Force, f ; Gas constant per mole, R ; Height, h ; Increment, Δ ; Length, l ; Mass, m ; Mean free path, λ , (λ) ; Micron, μ ; Millimicron, $\mu\mu$; Number (of terms, etc.), number of molecules, n ; Number of moles, N ; Pressure, p ; Pressure, osmotic, P ; Radius, r ; Ratio of circumference to diameter, π ; Reduced quantities: pressure, p_r , volume v_r , temperature, T_r , density, d_r ; Summation sign, Σ ; Surface tension, γ (σ); Time, t ; van der Waal's constants, a, b ; Variation sign, δ ; Velocity, u ; Velocity, angular, ω ; Velocity components in three directions, u, v, w ; Viscosity, η ; Volume (in general), v ; Volume, specific, v_s ; Volume, atomic, v_a ; Volume, molecular, v_m ; Weight, as gravitational force, w ; Work, W .

2. *General Chemistry*.—Atomic weight and gram-atomic weight, A ; Concentration (units not specified), C ; Equilibrium constant, K ; Mole fraction, x ; Molecular and gram-molecular weight, M ; van't Hoff coefficient, i ; Velocity coefficient, k .

3. *Heat and Thermo-Dynamics*.—Energy in general, (E) ; Entropy, Φ ; Intrinsic energy, U ; Latent heat, per grm., l ; Latent heat, per mole, L ; Mechanical equivalent of heat, J ; Molecular heat, S ; Molecular heat at constant pressure, S_p ; Molecular heat at constant volume, S_v ; Quantity of heat, Q ; Ratio of specific heats, $(=s_p/s_v)$ γ ; Specific heat, s ; Specific heat at constant pressure, s_p ; Specific heat at constant volume, s_v ; Temperature, Centigrade, t or t° (θ , when time occurs in the same expression); Temperature, absolute, T .

4. *Optics*.—Intensity of illumination, I (I_L); Refractive power, specific (Gladstone and Dale), r_G $[r_G]_D^\circ$; Refractive power, specific (Lorentz and Lorenz), r_L $[r_L]_D^\circ$; Refractive power, molecular, R_G R_L $[R_G]_D^\circ$ $[R_L]_D^\circ$; Angle of optical rotation, α ; Rotatory power, specific, $[\alpha]$; Rotatory power, molecular ($M[\alpha]$); Velocity of light, (v) ; Wave-length of light, λ .

5. *Electricity and Magnetism*.—Capacity (electric), C ; Charge, unitary (charge on an electron), e ; Conductivity (specific conductance) κ ; Conductivity equivalent, Λ ; Conductivity equivalent at different dilutions—vols. in litres containing 1 grm. equivalent, Λ_{10} , Λ_v , Λ_∞ ; Conductivity, equivalent of cation and anion, Λ_c , Λ_a ; Conductivity of specified ions, Λ_K , Λ_{Cl} ; Current, I ; Dielectric constant, K (as abbreviation, $D.C.$); Dissociation, electrolytic, degree of (degree of ionisation), α ; Electromotive force, E ; Faraday's constant, F ; Permeability, magnetic, μ (μ_p); Potential, single electrode, or decomposition potential of anion, ϵ K , Cl ; Potential measured against the hydrogen or calomel electrode respectively, which is taken as unity, ϵ_h , ϵ_c ; Quantity of electricity, Q ; Resistance, R (R_w); Susceptibility, magnetic, κ ;

Transport number of cation and of anion, n_c and n_a ; Velocity of cation and of anion in cm./sec. when the potential gradient is 1 volt/cm., U_c U_a ; Velocity of specified ions under unit potential gradient, U_K U_{Cr} .



IMPORTATION OF ORGANIC INTERMEDIATE PRODUCTS.

THE following letter has been received by the Registrar and Secretary of the Institute of Chemistry :

BOARD OF TRADE
(INDUSTRIES AND MANUFACTURES DEPARTMENT),
GREAT GEORGE STREET, LONDON, S.W. 1.
February 8, 1921.

SIR,—With further reference to your letter of January 28 regarding the Dyestuffs (Import Regulation) Act, 1920, I am directed by the Board of Trade to state that, whilst it is not possible to regard small quantities of organic intermediate products which may be required for research purposes as being outside the scope of the Act, the Board will be prepared to issue general licenses for the importation of such products to approved research institutions, covering periods of three months and limited only as to total quantities. This procedure will obviate the necessity for separate applications for a large number of small items, but it will be a condition of the issue of any general license that a detailed return shall be furnished, at the end of the three months during which the license is in operation, of the quantities of each product actually imported under it.—I am, sir,

Your obedient servant,

(Signed) PERCY ASHLEY.

THE REGISTRAR AND SECRETARY,
INSTITUTE OF CHEMISTRY OF
GREAT BRITAIN AND IRELAND,
30, RUSSELL SQUARE, W.C. 1.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

AN Ordinary Meeting of the Society was held on Wednesday, March 2, 1921, in the Chemical Society's Rooms, Burlington House. The President, Mr. Alfred Smetham, was in the Chair.

Certificates were read for the first time in favour of Messrs. Percy N. Mould and W. J. Wright. Certificates were read for the second time in favour of Messrs. Jules Cofman-Nicoresti, Walter K. Fletcher, William Singleton, Francis G. H. Tate, James Darnell Granger, Ph.D., F.I.C., Ed. B. Maxted, Ph.D. (Berlin), B.Sc., and Russell G. Pelly, F.I.C.

Mr. W. R. Schoeller, Ph.D., was elected a Member of the Society.

The following papers were read: "The Acidity of Ink and the Action of Bottle Glass on Ink," by C. Ainsworth Mitchell, M.A., F.I.C.; "The Detection of Adulteration in Butter by Means of the Melting Point of the Insoluble Volatile Acids," by George Van B. Gilmour, B.Sc., A.R.C.Sc.I., A.I.C.; and "Method and Apparatus for Routine Determination of Melting-Points of Fats and Fatty Acids," by S. H. Blichfeldt and T. Thornley.



OBITUARY NOTICES.

ÉMILE BOURQUELOT.

THE death of Émile Bourquelot, on January 31, deprives biological chemistry of one of its most active and distinguished workers. The long succession of papers upon the nature and behaviour of enzymes, which he published during a period of over forty years, and his crowning discovery of the reversibility of enzymic reactions have made his name familiar to chemists all over the world. His investigations on enzymes covered a very wide field, but were mainly concerned with the biology of plants. Many of his communications to French scientific societies had a direct bearing upon analytical chemistry, and were, therefore, abstracted in the ANALYST.

Bourquelot also took an active interest in pharmacy, and since 1900 had been General Secretary of the Société de Pharmacie de Paris, having served as its President in 1898.

EDITOR.

WILLIAM ODLING.

William Odling, who died on February 17, at Oxford, at the age of 92, was one of our earliest Honorary Members, and his death is, therefore, a loss not only to the scientific world, but also in a more intimate degree to our Society.

Odling was born in London in 1829, and began his professional career by taking a medical degree at the University of London, but he soon abandoned medicine for chemistry, and after being chemical lecturer at St. Bartholomew's Hospital, was appointed Professor at the Royal Institution in succession to Faraday. In 1872 he succeeded Sir B. Brodie as Waynflete Professor of Chemistry at Oxford, a position which he held for forty years. He was Secretary of the Chemical Society from 1856 to 1869, and was elected President in 1873, and he was one of the six Past-Presidents whose jubilee was celebrated in 1898.

Among Odling's contributions to theoretical chemistry were various communications developing Gerhardt's doctrine of chemical types, which led to the systematisation of organic chemistry, whilst his practical work included investigations of the compounds of platinum with ammonia and of the compounds of indium. He was also an authority upon the analysis of drinking water, and was one of the official analysts appointed to investigate the London water supply.

At the time of Odling's appointment to Oxford and for many years subsequently, the prevailing conception of the function of University professorships was that they were intended to provide the means and opportunity for research rather than to have an educational aim. The present writer well remembers the controversies that were continually arising over these conflicting views, and how slow was the evolution of the modern idea. Odling, accepting the older view, came but seldom into contact with the younger generation of Oxford chemists, and this was the more regrettable since, apart from the question of personal stimulus, he had as a lecturer the gift of making his subject interesting.

EDITOR.

JOHN CANNELL CAIN.

Dr. John Cannell Cain, whose death occurred suddenly at his residence in Brondesbury Park on Monday morning, January 31, had for the past fifteen years held the office of Editor of the Chemical Society's publications.

He was born at Edenfield, near Manchester, on September 28, 1871, and was the son of the Rev. Thomas Cain, of Stubbins, Lancashire. After receiving his early education at the Manchester Grammar School, he proceeded to the Owens College, where he took the degree of B.Sc. in the Honours School of Chemistry in 1892; thereafter he worked for a year at Tübingen, obtaining the degree of D.Sc. of that University in 1893. Early in his career Cain was drawn towards that field of organic chemistry to which he devoted his life, for in 1895 he began his practical acquaintance with the synthetic dyestuffs by joining Messrs. Levinsteins, Ltd., as a research chemist. In 1901, however, he again reverted to the teaching profession, and became Head of the Department of Physics and Chemistry in the Municipal Technical School at Bury in Lancashire, where he remained until 1904. It was

during this period that he started several important researches on subjects related to the organic dyestuffs, some of which he completed at Bury, leaving others to be finished at a later date elsewhere. It was from here, for example, that he published, with Frank Nicoll, the first three parts of the series on the rate of decomposition of diazo compounds, and as an outcome of this work he obtained the degree of D.Sc. in the University of Manchester in 1904. His appointment as Editor to the Chemical Society was made in 1906, after he had spent the two previous years as manager and head chemist to Messrs. Brook, Simpson, and Spiller, of London.

During his editorship Cain found time to complete many of the researches he had started at Bury, and also to publish several important textbooks. His earliest publication, that of the "Synthetic Dyestuffs," appeared in 1905 in collaboration with the writer, and this was followed in 1908 by the "Chemistry of the Diazo-Compounds," and in 1918 by a book on the "Manufacture of Intermediate Products." He also undertook to revise "Roscoe and Schorlemmer," and published Vol. I. shortly before his death.

During the war Cain placed his services at the disposal of the country, and at the end of 1914 was appointed by Lord Moulton as his first inspector of explosives. He also occupied the position of head chemist to the Dalton Works of British Dyes, Ltd., and was for a time Superintendent of H.M. Factory at Hackney Wick. There is no doubt that his strenuous work during this period was mainly responsible for his early death, because he had a serious breakdown in 1915 which left permanent weakness.

J. F. THORPE.



COMPOSITION OF THE HARROGATE MINERAL WATERS.

By WILLIAM LOWSON, B.Sc., F.I.C.

SINCE the discovery of the Tewit Well by Captain Slingsby in 1571, followed shortly afterwards by that of the Old Sulphur Well and the John's Well, the mineral waters of Harrogate have attracted considerable attention, and now some eighty springs are known, differing in type and quality. These may be divided into two main groups—viz., sulphur waters and iron waters—and each of these two groups is capable of further subdivision.

C. Fox-Strangways ("Memoirs of the Geological Survey," explanation of Sheet 62) gives an extensive bibliography of the literature up to the year 1908, and also an account of the geology of the district. In this he explains the existence of so many springs (no two of which are alike) issuing side by side, as due to the fact that Harrogate is situated at the eastern extremity of the great anticline which traverses this part of Yorkshire; the strata which are bent up by this anticline are the lower members of the Carboniferous series—the Millstone Grit, the shales and sandstones associated with the Harrogate Roadstone and the Mountain or Scar Limestone. The Millstone Grit of this district is for the most part a series of some

half-dozen beds of coarse grits, separated from one another by thick beds of shale. The series of rocks which include the Harrogate Roadstone consist in the upper part of dark blue shales, these being succeeded by thin bands of earthy limestone and chert. These cherty beds rest on dark blue shales containing sulphur, iron, and other chemical substances, and below these again there is a thin rubbly sandstone, also containing sulphur and iron, resting on other beds of shale.

The principal sulphur wells occur about a mile on either side of Harlow Hill, the highest point of the district.

Opinions differ as to whether the waters have a common origin, or whether each spring has its own independent source. The difference of level of adjacent wells favours the view of the separation of the several springs, and the temperatures indicate that they are neither superficial nor come from enormous depths. "Taking all things into consideration it seems the simplest and most probable theory that each spring has its own independent source, with which it is connected by a separate and distinct channel formed by the alternating series of pervious and impervious strata which connects Harrogate with the hilly regions to the west, for it is only from such a source as this that a large body of water could flow year after year, with such slight alteration both in quality and quantity; also that the chemical impregnation of the water is caused during its passage through these strata, and that it is from this source that the bases of all the salts are derived."

A general survey of these waters was undertaken some little time ago by Professor Arthur Smithells of this University, and in connection with this work the author carried out a series of analyses of the more important of them. From time to time in the past there have appeared in different journals accounts of investigations and analyses relating to these springs, and since the data thus recorded have found their way into books and publications of various kinds in many countries, it appears to be desirable to place on record the results of these more recent analyses. The work was commenced towards the end of the year 1912, and the figures given refer to samples collected during that and the two or three succeeding years, the delay in publication being occasioned by the war.

The results are recorded in terms of ionic concentrations.

Further information relating to the different waters is to be found in earlier publications; the following short notes, however, may be of interest: *Old Sulphur*.—A spring situated in the basement of the Old Pump Room; the water percolates through fissures in the Yoredale Rock Stratum, and flows into an artificial basin about $2\frac{1}{2}$ feet deep, and of a similar diameter. *Mild Sulphur*.—Occurs in close proximity to the Old Sulphur Spring, is much less concentrated, and varies considerably in strength from time to time. *Strong Montpellier*.—The well is about 15 feet in depth, and there is usually about 4 feet of water, which is removed by pumping. This water, as well as the Mild Montpellier, shows great variations in strength at different times. *Mild Montpellier*.—The well is about 20 feet in depth, and water accumulates to a depth of about 5 feet. *Magnesia*.—A surface spring occurring at the upper end of the Bogs Field, and fairly constant in composition. *Number 36*.—A surface spring at the lower end of the Bogs Field. *Harlow*.—This is the well commonly known as the Harlow Magnesia Well, and has a depth of $4\frac{1}{2}$ feet.

SUMMARY OF ANALYSES.

Parts per 100,000.

	Old Sulphur Well, Nov., 1912.	Mild Sul- phur Well, Nov., 1912.	Strong Montpellier, Dec., 1913.	Mild Mont- pellier, Apr., 1913.	Magnesia Well, Oct., 1913.	No. 36, Jan., 1913.	Harlow, July, 1915.	Beck- with, Aug., 1915.	Starbeck, Feb., 1914.
Sp. gr. ($\frac{15^{\circ}\text{C.}}{15^{\circ}\text{C.}}$) ...	1.0113	1.0056	1.0076	1.0068	1.0021	1.0033	1.0006	1.0004	1.0019
Total solids ...	1508.5	680.1	1003.3	874.0	258.15	415.7	52.2	45.08	222.3
Sodium ...	506.3	220.25	327.5	300.4	85.7	143.9	10.1	11.04	76.9
Potassium ...	6.5	3.85	4.56	4.51	1.68	2.43	0.66	0.56	0.94
Lithium ...	0.18	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Ammonium ...	1.68	1.16	1.27	0.51	0.29	0.78	—	—	0.27
Magnesium ...	18.24	11.26	15.44	15.49	3.10	4.5	3.62	1.60	2.70
Calcium ...	44.0	23.3	36.86	34.7	78.2	11.09	6.09	3.99	7.35
Strontium ...	Trace	Trace	Trace	Trace	Trace	Trace	—	—	—
Barium ...	6.04	—	0.47	4.9	3.36	1.88	—	—	0.89
Iron ...	—	—	—	—	—	—	—	—	—
Aluminium ...	—	—	—	—	—	—	—	Trace	—
Manganese ...	Trace	—	Trace	—	Trace	—	—	Trace	—
Chloride ...	879.1	382.5	578.0	499.9	134.5	221.2	4.30	3.00	103.5
Bromide ...	2.67	0.9	2.0	1.6	0.4	0.8	—	—	Trace
Iodide ...	Trace	—	—	Trace	—	—	—	—	—
Silicate (SiO_2) ...	1.20	0.76	1.04	1.11	1.62	1.08	1.21	1.20	1.77
Sulphate (SO_4) ...	—	11.14	5.73	0.43	0.53	1.65	4.0	—	7.2
Phosphate (PO_4) ...	—	—	—	—	—	—	—	Trace	—
Carbonate (CO_3) ...	59.7	53.0	54.9	80.8	67.1	61.4	61.5	46.6	66.15
Sulphur (as Sul- phide) ...	8.94	4.85	4.97	6.64	0.68	4.06	0.93	1.18	0.86

Parts per 100,000.

	Kissingen, June, 1914.	Chloride of Iron, Oct., 1914.	Alexandra Chalybeate, Dec., 1914.	Puro Chalybeate, Nov., 1914.	Tewit Well, Mar., 1915.	John's Well, Feb., 1915.	Alum Well, Apr., 1915.
Sp. gr. ($\frac{15^{\circ}\text{C.}}{15^{\circ}\text{C.}}$) ...	1.0071	1.0051	1.0027	1.0001	1.0004	1.0002	1.0028
Total solids ...	988.1	686.3	319.7	12.96	42.15	18.19	358.5
Sodium ...	293.1	207.8	110.4	0.86	1.91	1.46	118.1
Potassium ...	4.33	2.44	2.20	0.27	0.25	0.22	2.05
Lithium ...	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Ammonium ...	—	—	—	—	—	—	—
Magnesium ...	16.80	12.11	3.78	0.66	2.87	1.06	3.97
Calcium ...	41.2	29.44	10.41	1.54	5.60	2.49	10.83
Strontium ...	Trace	Trace	Trace	—	—	—	—
Barium ...	4.80	3.13	2.80	0.06	—	0.06	0.75
Iron ...	1.55	5.05	3.58	0.83	1.71	0.91	3.45
Aluminium ...	—	—	—	—	Trace	—	Trace
Manganese ...	Trace	0.07	Trace	Trace	Trace	0.02	Trace
Chloride ...	578.3	397.3	178.2	1.39	2.29	1.50	186.8
Bromide ...	1.0	1.0	1.0	—	Trace	—	—
Iodide ...	—	Trace	—	—	—	—	—
Silicate (SiO_2) ...	1.76	1.69	0.97	1.43	2.36	3.13	1.24
Sulphate (SO_4) ...	1.47	0.13	0.96	0.85	13.96	0.62	6.14
Phosphate (PO_4) ...	Trace	Trace	Trace	Trace	0.11	Trace	Trace
Carbonate (CO_3) ...	76.5	41.1	82.0	42.1	35.8	42.03	58.04

Beckwith.—A spring emerging at ground level. *Starbeck*.—The well is about 14 feet deep, and usually contains 2 or 3 feet of water, being fed by springs which emerge from the shales of the Millstone Grit. *Kissingen*.—The well is about 16 feet deep, and is fed by four bore holes sunk in the inclined stratum of shale, and each yielding a water of different composition, hence the water from this well shows considerable variations in strength from time to time. *Chloride of Iron*.—The well is about 15 feet in depth, and is fed by a bore hole 6 feet deep, penetrating the shale which at this point is almost vertical. *Alexandra Chalybeate and Pure Chalybeate*.—These are two surface springs, constantly flowing, and the latter yields the larger volume of water. *Tewit*.—The well is 7 feet in depth, and usually contains about 3 feet of water. *John's*.—A shallow well about 4 feet in depth. *Alum*.—A collection of very superficial water, which is found in the Bogs Field, surrounded by the Sulphur Wells. The yield is small, and for a period of eighty years the site was forgotten, being re-discovered in 1870.

In the case of the Old Sulphur Well it is interesting to note how closely the author's figures agree with those obtained by Thorpe in 1875, and a table of various analyses made during a range of some ninety years by different investigators is appended for comparison, the results here being expressed in terms of grains per gallon :

OLD SULPHUR WELL.

Grains per Gallon.

	West, 1828.	Hunter, 1880.	Hofmann, 1853.	Muspratt, 1867.	Davis, 1872.	Thorpe, 1875.	Lowson, 1912.
Total solids	1024·8	1016·0	1095·919	1108·78	1046·56	1047·013	1055·95
Soda	484·0	471·0	474·054	470·635	—	477·022	477·82
Potash	—	—	33·869	44·165	—	6·063	5·48
Lithia	—	—	—	Trace	—	0·266	0·19
Ammonia	—	—	—	—	—	0·328	1·05
Magnesia	14·7	18·0	23·446	27·392	—	23·839	21·18
Baryta	—	—	—	3·68	—	4·838	4·72
Chlorine	623·9	608·0	650·384	654·908	615·62	613·770	615·37
Bromine	—	—	Trace	Trace	—	1·985	1·87
Iodine	—	—	Trace	Trace	—	0·103	Trace
Silica	—	—	0·251	—	—	0·703	0·66
Sulphur	—	—	6·853	6·737	6·412	6·532	6·26
Sulphuric acid	Absent	Absent	0·101	Absent	—	Absent	Absent
Lime	39·8	43·5	48·243	46·233	—	38·697	43·12
S.G.	1023·24	—	1011·13	—	1011·16	1011·04	1011·28
Carbon dioxide	—	—	—	—	—	35·404	30·66
Free H ₂ S } cubic inches	—	—	5·31	7·01	—	10·16	—
Free CO ₂ } per gallon	—	—	22·03	25·55	—	40·10	—

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THE UNIVERSITY, LEEDS.



THE ACIDITY OF INK AND THE INFLUENCE OF BOTTLE GLASS UPON INK.

By C. AINSWORTH MITCHELL, M.A., F.I.C.

(Read at the Meeting, March 2, 1921.)

THE FUNCTION OF ACID IN INK.—The control of the acidity is one of the most important points in the manufacture of iron-gall inks, for it is the principal factor upon which the stability of the preparation depends. In a former communication (*ANALYST*, 1920, 45, 247) I have shown that in the oxidation of ink in the so-called "drying" process there is a gradual change from a soluble ferrous tannate first into colloidal and then into insoluble tannates of iron, and that this oxidation may be accelerated by the presence of certain catalysts. In the case of a mixture of a solution of ferrous sulphate and gall extract, or gallotannic acid, this change takes place fairly rapidly, and deposits and incrustations are formed in the liquid. By adding a certain proportion of strong acid, however, the oxidation process is arrested, and by a suitable regulation of the proportions of the mixture the ink is retained in the form of a stable solution or colloidal suspension until, when applied to the paper, the proportions become so altered by evaporation that the successive changes into the final insoluble resinous iron tannate take place.

AMOUNT OF ADDED ACID IN INK.—It is essential that the quantity of acid added should be kept within definite limits. If too little be added the ink will not acquire the desired stability, whilst if the amount be materially exceeded the ink will have an excessive corrosive action upon steel pens, and the dyestuff used as a provisional colour may even be bleached.

Ink-makers have gradually learned by experience to know the permissible variations of the acidity of their products, but unless this factor be chemically controlled unexpected troubles may occur.

The acids commonly used as stabilising agents are sulphuric, hydrochloric, and oxalic acids, preference being usually given to hydrochloric acid. In the German standard ink 2.5 parts of hydrogen chloride are added to 1,000 parts of ink. This formula has also been adopted for use as a comparative standard in the United States specifications for writing ink (*ANALYST*, 1921, 61). The British Government specifications, which were issued a few months ago, fix standards for the proportion of iron, but no reference is made to the acidity of the inks.

INKS WITHOUT ADDED ACID.—Several manufacturers prepare an ink to which no addition of strong acid is made. These consist essentially of iron gallate or of a complex iron gallate, for solutions of such salts when exposed to the air do not undergo the changes to which solutions of ferrous tannate are liable. A solution of gallic acid and ferrous sulphate is practically stable under conditions that will cause a solution of gallotannic acid and ferrous sulphate to yield deposits and incrustations. For example, I exposed a solution of 3 grms. of gallic acid and 2 grms. of ferrous sulphate to the air for thirty-five days and only obtained a deposit

of 0.036 grm., whilst the filtrate on further exposure for weeks yielded only a slight trace of deposit.

Hence a gallic acid ink has the advantage over an iron-gall ink of not requiring the addition of acid, and so of having little, if any, corrosive action upon a steel pen, although this point is not of importance in the case of fountain-pen inks, which are intended to be used with pen-nibs not attacked by dilute acid. On the other hand, the general opinion of practical inkmakers with whom I have discussed the subject is that gallic acid inks lack "body" and do not penetrate the fibres of the paper so readily as acidified gallotannic inks. The low proportion of solid matter in some of these inks is attributable to the small solubility of gallic acid in water (about 0.5 per cent. at 15° C.), which also limits the proportion of iron which can be introduced into the ink. Some of the oxyferrigallates are more soluble than ferrous gallate, but I am doubtful whether it would be possible to prepare a standard ink containing the 0.5 per cent. of iron required by the British Government specifications solely by the use of gallic acid.

The other question of the alleged inferiority in penetrating power of gallic acid inks could probably be definitely decided by comparative microtome tests.

EFFECT OF FERROUS SULPHATE ON THE ACIDITY.—The final composition of the ink depends to a considerable extent upon the copperas used in the manufacture, and variations in this salt may also have an influence on the acidity.

Theoretically, ferrous sulphate, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, contains 20.08 per cent. of iron. The presence of iron in excess of this amount is usually due to crystallisation of the salt at higher temperatures. Various commercial samples of copperas which I have examined during the last two or three years have contained the following amounts of iron: (1) 19.43; (2) 18.77; (3) 25.92; (4) 20.43; and (5) 19.36 per cent.

In nearly every instance the manufacturer's price was proportional to the amount of iron in the salt, and practical tests showed that an ink prepared from samples such as No. 2 would contain too little iron, if the calculation had been based on the use of a 20 per cent. copperas.

Commercial copperas has almost invariably a slight acid reaction. The acidity of the above-mentioned samples, in terms of *N*-alkali, varied from 0.11 to 0.37 c.c. per 1 grm. Obviously this acidity increases the amount of strong acid in the final ink, and accounts for the presence of such acid in inks to which no mineral acid has been added as such.

In estimating the acidity of copperas the ordinary indicators, such as litmus or phenolphthalein, are not of much use, since, unless added in very large quantity, they are bleached. Better results are obtained by titrating the solution without any indicator, the end-point being shown by the liquid turning bluish-green as soon as the precipitation of ferrous hydroxide begins. Houben's method (*Ber.*, 1919, 52, [B], 1623) of using ferrous sulphide as an indicator is not satisfactory.

Another way in which the ferrous sulphate may influence the acidity of the ink is through the formation of a basic salt. Copperas which has undergone superficial oxidation, with the formation of a coating of yellow basic sulphates, is technically termed "rusty," and is usually preferred by ink manufacturers, probably owing to the fact that the use of such salt accelerates the darkening process in the ink. Such

copperas may show pronounced acidity, and yet still neutralise added acid, so that an ink made from "rusty" copperas may show a different acidity from one prepared from the fresh green salt.

ESTIMATION OF STRONG ACID IN INK.—The presence of the blue dyestuff used as provisional colouring matter renders direct titration of ink very uncertain. Electro-metric titration would probably give satisfactory results, but the conditions would have to be worked out for each kind of ink. That is to say, the conditions for a hydrochloric acid ink would differ from those required by an oxalic acid ink, and in every case a method would have to be devised to distinguish between the strong acids and the weak organic acids in ink.

The only method hitherto described for estimating strong acids in ink is the somewhat crude one of immersing a weighed pen nib in a measured quantity of the ink, and estimating the amount of metal dissolved. This method, which I devised some years ago and published in my book on inks, has recently been included in the United States specifications (*supra*) without acknowledgment.

A much more accurate and rapid method is to distil 10 c.c. of the ink with an excess of a neutral solution of sodium acetate, and to titrate the acetic acid in the distillate. The objection to the process is that it requires at least three distillations of portions of about 100 c.c. each to expel practically the whole of the acetic acid, for Leeds (*ANALYST*, 1895, 20, 224) and subsequently Brode and Lange (*ANALYST*, 1909, 34, 157, 1909, 30) have shown that traces of free acetic acid are obstinately retained by organic matter. Distillation with xylene as a carrier, as suggested by Pickett (*J. Ind. Eng. Chem.*, 1920, 12, 570), does not offer any advantage in this case.

ESTIMATION OF THE TOTAL ACIDITY.—The method that I have found to be the most satisfactory means of estimating both the added acid and the weak organic acids in ink is to bleach the pigments with hydrogen peroxide as follows: Five c.c. of the ink are boiled for a few minutes with 10 c.c. of the ordinary 10 vol. hydrogen peroxide solution beneath a reflux condenser, the flask being continually shaken until a pale straw-coloured solution is obtained. This is cooled, diluted with three or four times its volume of water, and titrated with *N*-alkali solution, with phenolphthalein as indicator. A blank test is also made with 10 c.c. of hydrogen peroxide diluted with 10 c.c. of water, and the acidity deducted from the previous result.

I have proved by experiment that the hydrogen peroxide treatment does not oxidise any constituents of gall extract into acids, which would otherwise increase the acidity of the ink.

APPLICATION OF THE METHOD TO LOGWOOD INKS.—Hæmatoxylin, the colouring matter of logwood, is not bleached when boiled with hydrogen peroxide, and the method is therefore not directly applicable to logwood inks. Hence, in the case of such inks it is necessary to use the following modification of the method: Five c.c. of the ink are boiled with 20 c.c. of hydrogen peroxide solution and 2 c.c. of *N*-sodium hydroxide solution for about five minutes, until the liquid becomes transparent and pale. It is then cooled, diluted with a large volume (five or six times) of water, and the excess of alkali titrated with standard sulphuric acid, with Congo red as indicator (litmus or phenolphthalein being bleached). As before, a blank test

is made, to determine the correction to be applied for the acidity of the hydrogen peroxide.

No acids are formed when hæmatoxylin is boiled with the alkaline hydrogen peroxide, and sulphuric acid added to such a solution can be accounted for quantitatively after the treatment. In the case of Antoine's ink (No. 1 in the table), which is a logwood ink, it is interesting to note that the total acidity estimated by the hydrogen peroxide method is very slightly lower than the acidity due to strong acids, as found by the distillation method.

ACIDITY OF COMMERCIAL INKS.—The following results were obtained by means of the methods described with representative samples of commercial inks, the results in each case being expressed in terms of c.c. of *N*-alkali neutralised by 10 c.c. of ink.

Ink.				Total Acidity.	Strong Acids.	Weak Acids.
				c.c.	c.c.	c.c.
1.	Antoine, Violet black ...	...	...	1.60	1.76	nil
2.	Arnold, Blue-black ...	...	...	1.93	0.30	1.63
3.	Blackwood, "Record" ...	...	...	5.62	1.55	4.07
4.	Cochran's Blue-black (1912) (heavy deposit)	...	...	1.93	0.60	1.33
5.	" " Acid-free ink ...	...	...	3.10	0.01	3.09
6.	Field's Blue-black, old ...	...	...	2.66	—	—
7.	" " " " , new ...	...	...	2.99	0.17	2.82
8.	Hollidge, Blue-black (heavy deposit)	...	...	7.0	0.40	6.60
9.	Mordan's "Azuryte" ...	...	...	4.06	0.98	3.08
10.	Morrell's Blue-black (faded, heavy deposit)	...	...	1.92	0.07	1.85
11.	Stephen's Blue-black ...	...	...	3.83	2.46	1.37
12.	"Swan" ...	...	...	3.42	1.82	1.60
13.	Typical Blue-black ink (deposit), April ...	...	...	3.20		
(a)	" " (faded), August ...	...	...	0.15		
(b)	" " (alkaline), January ...	...	...	-0.30	nil	
14.	Tarling, Blue-black ...	...	...	3.85	1.35	2.50
15.	Webster "Diamine ink" (old, heavy deposit)	...	...	0.48	0.08	0.40
16.	" " " " (new) ...	...	...	3.68	0.18	3.50

ACIDITY CONTROL OF THE MANUFACTURING PROCESS.—The hydrogen peroxide method affords a simple method of controlling the correctness of the manufacturing process. For example, in the case of an ordinary iron-gall or gallic acid ink prepared by a specified formula the total acidity remains fairly constant from month to month. Any pronounced variation from this figure will point either to some deviation from the formula, or to the action of some external influence.

Last year complaints were received by different manufacturers of ink of the occurrence of deposits in the bottles, and in some of the worst cases the ink itself had faded. The composition of the ink was in most cases normal, with the exception of the acidity being somewhat lower than usual, and it therefore appeared as though the ink had been sent out insufficiently matured, so that a deposit which should have formed in the vats had formed in the bottles.

It was not until I examined a bottle in which the colour of the ink had changed to a dirty brown that I found that there was something radically wrong. In that case the total acidity by the peroxide method was only 1.5 c.c., or rather less than half the normal value. As there was no evidence of contamination with an alkaline salt, I tested the glass of the bottle itself, and this gave me the solution of the problem. It was not the ink, but the bottle which was at fault.

INFLUENCE OF ALKALINE GLASS.—When cold distilled water was placed in the empty bottles, after thorough cleaning, or when the bottles were immersed in water, alkali was rapidly dissolved, and, after about five minutes, the water showed a pronounced alkaline reaction towards phenolphthalein.

Two series of tests were made with two new bottles of the same consignment. Each was filled with water, and 10 c.c. portions were titrated with *N*-sulphuric acid at subsequent periods, with the following results: I. After 4 hours, 0.47 c.c.; after 24 hours, 0.94 c.c.; after 48 hours, 0.94 c.c.; after 72 hours, 0.94 c.c.; and after 9 days, 0.47 c.c. II. After 3 days, 0.71 c.c.; 13 days, 0.75 c.c.; 20 days, 0.66 c.c.; and 24 days, 0.14 c.c.

It appeared, therefore, that the reaction was retrogressive, the alkali first liberated by the water from the sodium silicate or the alkali carbonate dissolved again entering into combination with other constituents of the glass.

An analogous test was made by placing 18.9 c.c. of *N*-sulphuric acid in bottle No. 1. After eleven days the acidity had fallen to 12.8 c.c.

The same effect is produced by ink, as is seen by the results given by the sample No. 13, in which the change was followed from an acidity of 3.2 c.c. to an alkalinity of 0.30 c.c.

Nor does ink free from added acid fare any better when placed in these bottles. For instance, a sample from a bottle of Field's ink, the bulk of which is still quite free from any deposit, produced a thick incrustation on the sides of one of these bottles within three days, whilst its total acidity, as determined by the hydrogen peroxide method, fell from 2.99 c.c. to 0.35 c.c. in sixty days, and the colour changed from bright blue to brownish purple.

The explanation offered by the makers of these glass bottles was that owing to their being unable to obtain supplies of salt-cake they had used soda-ash in the manufacture of this glass, and had thus introduced an excess of alkali. Incidentally they suggested that probably all bottle glass would be found to be more or less alkaline. Although the term "alkalinity" may be regarded as relative in this connection, some bottle glass, at all events, is practically neutral, for I have kept standard sulphuric acid for several years in a bottle without its showing more than the slightest depreciation.

At the same time, it is quite possible that the cheaper kinds of glass bottles, such as are used for ink, may not infrequently yield a considerable proportion of free alkali to any liquid placed in them, and that this may be the hitherto unsuspected reason for the occasional deterioration of ink in bottle.

This was borne out by an experiment with the bottle which had contained the faded, decomposed Morrell's ink (No. 10). After thorough washing, the bottle was filled with distilled water, and the liquid tested daily. For five days it remained

neutral to phenolphthalein, but on the sixth day gave a very faint pink coloration, and on the seventh day a distinct alkaline reaction. Hence, although this glass was much less alkaline than that of the bottles mentioned, it seems reasonable to conclude that in this instance, also, it was the cause of the instability of the ink.

The glass of the very alkaline bottles was distinctly vitreous in character, but this less alkaline glass was much clearer, and it is possible that a relationship might be established between the alkalinity and the transparency of glass.

If the alkalinity of bottle glass be as common as these glass manufacturers suggest, it may have an important bearing upon other products. For instance, the 33 per cent. acetic acid of the British Pharmacopœia would soon be reduced below the standard strength if sent out in bottles of this type, whilst such glass might cause the precipitation of quinine from pharmacopœial preparations of quinine salts.

In chemical work my chief difficulties have been caused, not by the alkalinity of glass bottles, but by the solution of iron from the glass by acids, this being particularly pronounced in the case of Winchester quart bottles. After some time hydrochloric acid has become quite yellow from this cause, and has given a strong Prussian blue reaction.

In conclusion, I wish to thank Messrs. Mabie, Todd and Co. for permission to bring before you such of these results as were obtained in the routine examination of their inks.

DISCUSSION.

DR. R. D. BAIN asked whether the author did not find that the action of hydrogen peroxide on some inks tended to increase the acid. *Hæmatoxylin*, for instance, when oxidised under suitable conditions, gave formic acid. Might it not be possible that intermediate acids were formed during the boiling with hydrogen peroxide? The excessive alkali shown in some of the bottles might be due, not so much to the excess of alkali in the glass, as to the degree of resistance of the glass to corrosive action. The proportion of saltcake to soda ash in the constitution of a glass had an influence upon the skin for resisting purposes. But the substitution of saltcake for soda-ash, or *vice versa*, did not have any material influence on the final alkalinity of the glass. It was the proportion of alkali to lime that could change the alkalinity of the glass; and this was largely a question of expediency in the glass bottle trade. The introduction of machine-made bottles into England had necessitated a change in constitution, and "sweeter" glass was required for machine-made bottles than for hand-made bottles.

He wished to draw the author's attention to the work of Wood, Sand, and Law (*J. Soc. Chem. Ind.*, 1911) where these investigators used the electrometric titration method for the investigation of dark tanning liquids. It appeared to him that the determination of acidity by means of the concentration of H-ions would not only render one independent of the colour of the liquid for the final point, but might possibly give a more rapid estimation.

MR. G. RUDD THOMPSON inquired whether, in view of the importance of an acid-free copperas to ink manufacturers, it would not be possible to use ferrous ammonium

sulphate instead of the ordinary ferrous sulphate. There would be no difficulty in providing supplies of the double salt.

Dr. G. MONIER-WILLIAMS emphasised the advantages of electrometric methods of titration. Since the acidity due to strong acids in the ink appeared to be the important point, he wished to know what advantage was gained by determining the acidity due to the weak organic acids.

Mr. E. T. BREWIS remarked that it was fairly well known that the "metal" of which such cheap bottles as those exhibited were made was partially soluble in distilled water, yielding an alkaline solution. Very cheap bottles were made from "cullet," and sometimes, as in the case of Yorkshire cullet, the pot was "flushed" with soda-ash to lower the fusion point of the metal. The bubbles and flaws in the bottles passed round were due to imperfect fusion, and the alkalinity of the glass might be due to an attempt to rectify those faults by the addition of soda rather than by expenditure of fuel. It might be of interest to mention that the greatest care was necessary in selecting the glass tubing used for making "ampoules" for sterile sera and alkaloidal solutions, the slightest trace of alkalinity causing such solutions to deteriorate on storage.

Mr. T. F. HARVEY pointed out that methyl red could be used as a satisfactory indicator for the alkalimetric titration of ferrous sulphate solutions.

Mr. MITCHELL, replying to Dr. Bain, remarked that the difficulty with regard to electrometric titration was that a separate standardisation would be necessary for each kind of ink. For any given kind of ink, however, it would probably be a very satisfactory method of factory control. Within the limits of his experience he had not found that oxidation of hæmatoxylin occurred in the peroxide method, and a small addition of sulphuric acid to a logwood solution could be estimated quantitatively. The advantage of estimating the total acidity of the ink was that it served as a control of the factory operations, any variation from the formula being immediately indicated. Referring to Mr. Thompson's suggestion of the use of ferrous ammonium sulphate in place of ferrous sulphate, he said that he had already made a series of experiments with the former salt, the results of which had indicated that the double salt would be a satisfactory substitute for ordinary copperas. A slight variation in the acidity of ferrous sulphate was not a matter of great importance for an ordinary acidified iron-gall ink, but in the case of non-corrosive gallic acid inks, it was essential that no strong mineral acid should be present.



ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Iron as the Cause of the Formaldehyde and Diphenylamine Reaction of Milk. F. Reiss. (*Zeitsch. Unters. Nahr. Genussm.*, 1921, 41, 26-29.)—The presence of iron in milk causes the latter to yield a reaction with diphenylamine sulphuric acid similar to that given by nitrates or nitrites. Milk may become contaminated with iron when it is kept in rusty churns or cans, and if iron be detected by the ferrocyanide reaction the test for nitrates should be applied to the milk serum (obtained by allowing the milk to curdle spontaneously) and not to the milk itself.

W. P. S.

Mustard Oil as a Milk Preservative. P. Post. (*Pharm. Weekblad*, 1921, 58, 131-138.)—Mustard oil may be added to milk, in the proportion of 20 drops per litre, as a preserving agent. The treatment has no influence on determinations of specific gravity, fat, acidity, total solids, ash, chlorides, nitrates, preservatives, specific gravity and refraction of the serum, or the polarisation. Addition of mustard oil depresses the freezing-point; with a high fat content the error is small, and it may be almost entirely eliminated by one agitation with 10 per cent. of sesamé oil. In lactose estimations the mustard oil must first be removed by shaking the milk three times with petroleum spirit, and for the determination of albumin the sample must be evaporated to half its bulk. Catalase is decomposed by mustard oil.

W. J. W.

Coffee Extracts. G. S. Ponce and J. R. Palma. (*Bol. Lab. Municipal de Guayaquil*, 1920, 1, 65.)—Twenty-four samples of genuine coffee extract contained from 9.04 to 11.91 per cent. of water (average 10.62); 82.69 to 85.05 per cent. of organic substances (average 83.68); and 4.92 to 6.66 per cent. of ash (average 5.61 per cent.).

Composition of Chicha. C. D. Andrade. (*Bol. Lab. Municipal de Guayaquil*, 1920, 1, 71-74.)—Chicha is a fermented beverage, prepared from maize, which is largely used by the working classes in Guayaquil. In the course of its manufacture toxic nitrogenous putrefaction products are formed, and to these are attributed the poisonous effects of the drink upon certain individuals. Ten samples had the following composition: Acids (as tartaric acid), 4.15 to 8.70; alcohol, 26.5 to 54.63; sugars (dextrose and invert sugar), 7.65 to 9.17; nitrogenous substances, 5.98 to 6.40; glycerol, 4.05 to 4.70; and mineral matter (ash), 2.59 to 2.87 grms. per litre.

Estimation of Sugar in Wine. W. Fresenius and L. Grünhut. (*Zeitsch. anal. Chem.*, 1920, 59, 415-457.)—With non-hydrolysed wines of acid reaction, lead acetate may be employed as a clearing agent, if not in excess. In the case of wines

which have been hydrolysed by hydrogen chloride, and are acid, lead acetate may be used for precipitation of the chlorine, but other impurities cannot be removed. After neutralisation these can indeed be precipitated by excess of lead acetate, but a loss of sugar then results in presence of chlorides. It is therefore preferable either to substitute bone black for the lead acetate, or, if the latter be used, to precipitate the chlorine ions first by means of another reagent, such as silver nitrate. W. J. W.

Preparation of Rhamnose. C. F. Walton, jnr. (*J. Amer. Chem. Soc.*, 1921, 43, 127-131.)—Rhamnose may be prepared conveniently in the laboratory from "flavin," a commercial product rich in quercitrin, the flavin being hydrolysed by boiling it for thirty minutes with 10 parts of water and 0.5 per cent. of sulphuric acid (calculated on the flavin plus water), the residue washed free from sugar, and the combined filtrates neutralised with barium carbonate, decolorised, and concentrated under diminished pressure until containing about 40 per cent. of solids. The inorganic impurities are then precipitated by the addition of alcohol, and the filtrate concentrated until it contains 70 to 80 per cent. of solids, and allowed to crystallise; the yield of rhamnose is 20 to 25 per cent. of the flavin taken. T. H. P.

Volumetric Method for the Estimation of Lactose by Alkaline Potassium Permanganate. F. T. Adriano. (*Philippine J. Sci.*, 1920, 17, 213-220.)—The following method is described for the estimation of lactose by oxidation with alkaline potassium permanganate solution. Twenty-five c.c. of the milk are mixed with about 400 c.c. of water, 10 c.c. of Fehling's copper sulphate solution are added, the mixture is nearly neutralised with sodium hydroxide solution, diluted to 500 c.c., and filtered. Ten c.c. of the filtrate are mixed with 50 c.c. of $\frac{N}{10}$ permanganate solution and 25 c.c. of 0.848 per cent. sodium carbonate solution, the mixture is diluted to 100 c.c., and heated, the temperature being raised to 95° C. in two minutes and maintained for a further two minutes. Twenty-five c.c. of 30 per cent. sulphuric acid are added and $\frac{N}{10}$ oxalic acid is introduced until the solution is clear; the excess of oxalic acid is then titrated with the permanganate solution. The amount of permanganate used for the actual oxidation is a measure of the quantity of lactose present, this quantity being found by reference to the table given below:

Lactose.	$\frac{N}{10}$ KMnO ₄ .	Lactose.	$\frac{N}{10}$ KMnO ₄ .	Lactose.	$\frac{N}{10}$ KMnO ₄ .	Lactose.	$\frac{N}{10}$ KMnO ₄ .
Mgrms.	C.c.	Mgrms.	C.c.	Mgrms.	C.c.	Mgrms.	C.c.
1	2.51	11	12.30	21	23.63	31	34.65
2	3.21	12	13.80	22	24.87	32	35.69
3	4.04	13	15.41	23	25.92	33	36.16
4	4.39	14	16.72	24	26.67	34	37.26
5	5.47	15	17.85	25	28.10	35	38.26
6	6.52	16	19.04	26	28.39	36	38.64
7	7.69	17	20.16	27	29.50	37	39.28
8	9.13	18	20.96	28	30.60	38	40.16
9	9.98	19	21.50	29	31.49	39	40.56
10	11.37	20	22.89	30	32.55	40	41.47

The results obtained are concordant, but slightly lower than those obtained by the Soxhlet method.

W. P. S.

Composition of Palm Kernels from Madagascar. G. Clot. (*Chem. Abstracts*, 1921, 15, 184, from *Mat. grasses*, 1920, 12, 5661-5662.)—*Hyphæne shatan* seeds gave the following analytical results: Moisture, 12.10; fat, 8.08; total ash, 2.39; soluble ash, 1.01; cellulose, 71.48; and nitrogenous substances, 5.95 per cent. The oil derived from the seeds gave an iodine value of 21.9, and saponification value 245.3; and hydrolysis of the cellulose yielded 24.7 per cent. of mannose and 9.65 per cent. of dextrose. *Borassus Madagascariensis* seeds gave: Moisture, 10.25; fat, 0.51; total ash, 1.74; soluble ash, 1.20; cellulose, 82.66; and nitrogenous substances, 4.84 per cent. Hydrolysis of the cellulose yielded 36.36 per cent. of mannose and 8.8 per cent. of dextrose.

T. J. W.

Analysis of Otoba Butter. W. F. Baughman, G. S. Jamieson, and D. H. Brauns. (*J. Amer. Chem. Soc.*, 1921, 43, 199-204.)—The sample of otoba butter (from *Myristica otoba*) examined was brown and of disagreeable odour. It contained 9.3 per cent. of essential oil, 20.4 of other unsaponifiable matter, and 67.6 of fatty acids, and its constants were: Sp. gr., 0.9293 at 20°/20° C.; refractive index, 1.4710 at 40° C.; iodine number (Hanus), 54.0; saponification value, 185.0; and m.-pt., 34.0° C. The essential oil, separated by steam distillation, had a slight odour and a yellow colour, the latter fading in the light; it consisted chiefly of sesquiterpenes, and had density at 20° C. 0.89067, $[\alpha]_D^{20} = -32^\circ$ and $n_D^{20} 1.4180$. The glycerides present in the butter were those of lauric (15.1 per cent.), myristic (52.2), palmitic (0.2), and oleic acids (3.9); and the unsaponifiable constituents, besides the essential oil, consisted of a viscous yellow mass (11.0 per cent.) and of the isomeric otobite and iso-otobite, $C_{20}H_{20}O_4$ (9.4 per cent. together). Otobite (m.-pt., 137-138° C.) and iso-otobite (m.-pt., 106-108° C.) both give with concentrated sulphuric acid a pink coloration, which becomes very intense on standing and persists for days. Addition of several drops of concentrated sulphuric acid to glacial acetic acid solution of either compound (phytosterol test) gradually produces a pink coloration, which turns somewhat purple after some hours and persists for some days. Attempts to obtain acetyl derivatives or to methylate the compounds with methyl sulphate were unsuccessful, whilst treatment with alcoholic potassium hydroxide solution and fusion with potassium hydroxide were without effect. Otobite contains one methoxyl group in the molecule, but iso-otobite none. Both compounds form pentabromides, $C_{20}H_{20}O_4Br_5$, (m.-pt., 190° C. and 191° C. respectively).

T. H. P.

Grape Seed Oil. E. Klinger. (*Chem. Abstracts*, 1921, 15, 181; from *Farben-Zeit.*, 1920, 26, 6.)—Examination of a pure sample of grape-seed oil gave the following results: Sp. gr. 0.925; saponification value, 192; iodine value, 130.9; whilst the fatty acids gave neutralisation value, 199.5; iodine value, 133.5; m.-pt., 23°-25° C.; and solidifying-point, 18°-20° C. The oil dried to a sticky film in four days, and in a shorter time when mixed with metallic driers. The oxygen absorption was 7 per cent. in four days, whilst a control, using linseed oil, showed 14.7 per cent. On being

heated to 300° C. the oil developed "body" more rapidly than linseed oil, and in a few hours became a gummy mass.

T. J. W.

New Hydroxystearic Acid from Hardened Castor Oil. H. Thoms and W. Deckert. (*Ber. Deut. Pharm. Ges.*, 1921, 31, 20-26.)—From a sample of hardened castor oil (m.-pt., 80° C.; iodine value, 12) boiling alcohol extracts 1-12 hydroxystearic acid, $\text{CH}_3\cdot[\text{CH}_2]_6\cdot\text{CH}(\text{OH})\cdot[\text{CH}_2]_{10}\cdot\text{CO}_2\text{H}$ (m.-pt., 83° C.; iodine value, 0). The ethyl ester of the acid was prepared, as well as the anilide and *o*-toluidide, the corresponding ketostearic acid formed on oxidation, and other derivatives.

T. H. P.

Bombay Mace. (*U.S. Dept. Agric. Bureau of Chemistry, Service and Regulatory Announcements*, No. 26, 21-22.)—Although "Bombay mace" closely resembles true mace in appearance, and is botanically a mace, it is practically devoid of flavour, and is useless as a spice. Its use in spices is merely as a filler and adulterant, and the Bureau of Chemistry is of the opinion that it should not be used as an ingredient of spices, even when these are labelled to show its presence.

R. G. P.

Changes produced in Wheat by Tempering. E. L. Tague. (*J. Agric. Research*, 1920, 20, 271-275.)—"Tempering," also known as "dampening" or "conditioning," consists in mixing water with the wheat and allowing the mixture to stand for some time before grinding. This treatment toughens the bran coat, produces an increased yield of flour, and enables better bread to be made. Several kinds of tempered wheat were ground, and a quantity of the flour equivalent to 50 grms. of dry material was added to ten times its weight of water at 40° C., 2 c.c. of toluene being added as preservative. The mixture was well shaken and maintained at 40° C. for two hours, with occasional shaking, after which it was whirled in a centrifuge for five minutes, the liquid filtered, and the filtrate examined. Tempering at 20° C. produced a slightly higher P_{11} value and a slight increase in the total acidity, water-soluble phosphorus, and "titratable nitrogen" (c.c. of $\frac{N}{10}$ NaOH required to titrate 10 grms. of flour), and the increase was still more pronounced at 40° C. The chemical changes increased with an increase in the time of tempering, but were detrimental after a period of forty-eight hours.

T. J. W.

Bleached Flour in U.S.A. (*U.S. Dept. Agric. Bureau of Chemistry, Service and Regulatory Announcements*, No. 26, 21.)—The Bureau regards as adulterated flour bleached by any process, if the bleaching has reduced the quality or strength or has concealed damage or inferiority. Bleached flour may, however, be shipped within the jurisdiction of the U.S.A. Food and Drugs Act if it satisfies the above requirements, provided that it is branded to indicate that it has been bleached. No action will be taken at the present time on the ground that bleaching introduces into the flour a substance which may be injurious to health, provided, as a result of bleaching, there is not introduced such a quantity of the bleaching agent as may render the flour injurious as indicated in a decision of the Supreme Court.

R. G. P.

Action of Halogen Acids on Oil of Juniper. R. Huerre. (*J. Pharm. Chim.*, 1921, **23**, 81-89.)—The crystalline compounds, obtained by treatment of oil of juniper with halogen acids, consist entirely of hydrochloride, hydrobromide, and hydriodide of cadinene. Treatment with acetic acid saturated with hydrogen iodide yielded, with three samples, 54 and 69 per cent.; 37.75 and 36.20 per cent.; and 37.50 and 51.25 per cent. of crystals. The relative amounts obtained with hydrogen iodide, bromide, and chloride were, in the case of one sample, 54 and 69, 24, and 12.40 per cent.; and with another sample, 44, 28, and 18.15 per cent. The respective yields obtained from equal quantities of cadinene were 18.15 per cent. of hydrochloride, 23.98 per cent. of hydrobromide, and 30.14 per cent. of hydriodide. An approximate estimate of the amount of cadinene (laevorotatory) in oil of juniper may be obtained from the yield of hydriodide: $C_{15}H_{24} \cdot 2HI$. This may attain a maximum of 30.59 per cent., but the average figure is 21.11 per cent. W. J. W.

Estimation of Thymol : Spanish Oil of Thyme. H. Mastbaum. (*Chem. Zeit.*, 1921, **45**, 18-19.)—Three successive extractions with 100, 80, and 50 c.c. of 5 per cent. sodium hydroxide solution, remove the phenols (thymol and carvacrol) from 25 grms. of the sample. The extracts are made up to 250 c.c. and filtered; 200 c.c. are rendered acid and cooled, and the oily layer is separated and weighed in a tared beaker; on stirring, the crystallisation of the thymol usually begins without inoculation. The crystals are collected on a weighed filter, pressed between paper, and weighed. Eight samples of Spanish oil of thyme had: Sp. gr. 0.9444 to 0.9747 at 15°/15° C., and contained 55 to 67.5 per cent. of phenols. O. F. M.

Estimation of Thymol in the Volatile Oil of *Mosla japonica*. T. Kariyone and K. Atsumi. (*J. Pharm. Chim.*, 1921, **23**, 148-149.)—The volatile oil of *Mosla japonica* contains *p*-cymene, thymol, and another phenol, probably carvacrol, and is the most important Japanese source of thymol. For the estimation of thymol, 10 c.c. of oil are dissolved in 40 c.c. of petroleum spirit, and extracted by shaking four times successively with 10 c.c. of sodium hydroxide solution 5 per cent.; the phenols are liberated from the alkaline liquid by acidification with 30 c.c. of hydrochloric acid (15 per cent.) and crystallised by cooling and, if necessary, by adding a crystal of thymol. The crystals are drained and, after washing with 20 c.c. of dilute alcohol (10 per cent.) and then with 30 c.c. of cold water, are dissolved in sodium hydroxide solution (5 per cent.), and made up to 500 c.c. The thymol in 10 c.c. of this liquid is estimated iodimetrically. If more than 32 per cent. of liquid phenols be present in the mixed phenols the method is useless, but where less than 9 per cent. is present (as in the oil in question) the error of determination does not exceed 2 per cent. R. G. P.

Extraction of Thymol and Carvacrol from Alkaline Solutions. D. C. L. Sherck. (*Amer. J. Pharm.*, 1921, **93**, 8-18.)—Both phenols are extracted in the free state from alkaline solution by shaking with ether, heptane, benzene, carbon tetrachloride, or carbon disulphide. The extraction of 50 c.c. of 5 per cent. sodium hydroxide solution containing 7.46 grms. of thymol with three successive 50 c.c.

portions of solvent gave the following percentages of the original thymol extracted: Ether 99.7, heptane 29.9, benzene 67.9, carbon tetrachloride 52.6, and carbon disulphide 66.2. Thymol recovered by the use of carbon disulphide was of more satisfactory appearance than when other solvents were employed. Increasing the concentration of sodium hydroxide in the aqueous solution has no effect upon the ether extraction, and but little effect when benzene or carbon disulphide is used, but the results with heptane and carbon tetrachloride are considerably affected. Thymol showed considerable deliquescence to ether vapour, but not to the vapours of the other solvents employed.

T. J. W.

Constituents of "Shê-chuang-tzu" (*Selinum Monnieri* L.) M. Nakao. (*J. Pharm. Chim.*, 1921, 23, 141-142.)—This drug, known in Japan as "dsha-sho-shi" and composed of the fruits of *Selinum Monnieri* L., is held in repute in China as an aphrodisiac and as a sedative. On steam distillation it yielded 1.3 per cent. of a yellowish limpid volatile oil with a turpentine-like odour; sp. gr. at 15°/15° C., 0.896; $[\alpha]_D^{20}$ -42.7°; acid value, 1.63; saponification value, 23.62; and saponification value after acetylation, 51.35. Potassium hydroxide and bisulphite solutions did not dissolve any of the oil. The following fractions were separated: (1) 154-157° C., $[\alpha]_D^{20}$ -55.42° contained *l*-pinene; (2) 157-160° C. contained camphene; (3) 200-250° C., $[\alpha]_D^{20}$ -33.14°; this consisted principally of an ester having a saponification value of 229 and yielding *l*-borneol and iso-valeric acid. The fractions of high b.-pt. gave a small quantity of crystals not identified. Phellandrene, cineol, and terpineol were not present.

R. G. P.

***Polygonum Bistorta* as a Substitute for *Krameria triandra*.** Ruiz and Pavon. (*Chem. Abstracts*, 1921, 15, 144, from *J. Pharm. Belg.*, 1920, 2, 876-878.)—Analysis of *Polygonum* gave the following results: Water, 6.3; ash, 4.35; tannic acid, 18.25; reducing sugars, 2.13; gums, 2.23; and starch, cellulose, and resin, 66.74 per cent. When tested with ammonium molybdate all the tissues were found to contain tannin, but the amount was greatest in the cortical parenchyma. The authors conclude that this plant is a satisfactory and economical substitute for *krameria*.

T. J. W.

Peru Balsam and its Adulteration. L. van Itallie. (*Amer. J. Pharm.*, 1921, 93, 24-26.)—Peru balsam is frequently adulterated by the addition of benzyl benzoate and other esters resembling the natural balsam in odour and appearance. Such adulteration may be detected by the following tests even when the usual analytical values are normal: (1) One drop of the balsam is heated with 0.1 gm. of resorcinol and ten drops of sulphuric acid, cooled, diluted with water, and treated with excess of sodium hydroxide solution. Genuine samples yield only a weak, green fluorescence, but when adulterated an intense green due to fluorescein is formed; (2) One gm. of balsam is dissolved in ether, the liquid shaken with 2 per cent. sodium hydroxide solution, and the alkaline extract acidified after separation. The precipitated resins are dissolved in ether, and the solution gently poured on to the surface of sulphuric acid, when adulteration is shown by the formation of a green or

blue layer. A commercial sample of *Balsamum peruvianum syntheticum* yielded normal values for the usual constants, but gave positive results with the above tests, whilst the addition of five drops of the sample to 8 c.c. of petroleum spirit produced a white precipitate, and a mixture of 1 vol. of carbon disulphide and 3 vols. of the balsam produced a jelly.

T. J. W.

Remijin, an Inferior Japanese Substitute for Optochin (Ethylhydrocupreine). C. A. Rojahn. (*Ber. Deut. Pharm. Ges.*, 1921, **31**, 37-38.)—Examination of remijin, made by the Sankyo Company, Muromachi, Tokyo, and described as ethylhydrocupreine, gave the following results: It was a light-grey powder insoluble in water, sintering at about 105° C. (optochin, 122° C.), m.-pt. 130 to 140° C. (optochin, 126 to 130° C.); loss on drying, 2·7 per cent.; ash, 0·27 per cent.; ethoxyl, 8·94 per cent., corresponding with 67·5 per cent. of the ethylated base; ethereal extract, 65·41 per cent.; and non-ethylated phenolic base, 30·85 per cent. Remijin hydrochloride, a yellowish-white crystalline powder from the same source, sinters at about 60° C., m.-pt. about 105° C. (optochin hydrochloride, 252 to 254° C.); loss on drying, 2·6 per cent.; ash, 0·33 per cent.; ethoxyl, 7·3 per cent., corresponding with 60·9 per cent. of optochin hydrochloride, and ethereal extract of the product rendered alkaline, 59·4 per cent.; non-ethylated phenolic base, 35·4 per cent. Thus, about one-third of the products consists of hydrocupreine, which differs from ethylhydrocupreine physiologically.

T. H. P.

New Rapid Method for the Estimation of Morphine in Opium and its Preparations. T. Ugarte. (*J. Pharm. Chim.*, 1921, **23**, 129-131.)—One grm. of powdered opium is warmed with 10 c.c. of alcohol (67 per cent.) on a water-bath, the clear liquid decanted through a filter, and the insoluble residue treated three times with 10 c.c. of alcohol. The total filtrate is evaporated to dryness in a dish on the water-bath and heated to 100° C. for fifteen minutes to oxidise and render the resins insoluble; the residue is then extracted successively four times with 5 c.c. of cold water (a flat-ended rod being used to break up the residue), and the liquid filtered and evaporated to dryness. The residue is taken up in 2 c.c. of water saturated with morphine, and 3 c.c. of dilute ammonia saturated with morphine are added (9·1 c.c. of ammonia of sp. gr. 0·925 diluted to 100 c.c.); 10 c.c. of ether are next added, and the liquid agitated so as to form an emulsion two or three times, the rapid evaporation of the ether at this stage lowering the temperature to within 2 to 3 degrees above zero. After the addition of another 20 c.c. of ether, the mixture is shaken and allowed to stand for thirty minutes, when the morphine crystallises in small granules on the bottom and sides of the beaker. The ethereal layer is decanted through counterpoised filters, and the morphine is treated with a further 10 c.c. of ether, transferred to the filter by means of a rubber-tipped rod and a jet of water saturated with morphine and ether, and is finally dried at 100° to 105° C. and weighed. In the case of opium preparations (*e.g.*, tinctures), 10 c.c. are evaporated to dryness and heated at 100° C. for fifteen minutes to oxidise the resins, and then treated as above.

R. G. P.

Identification of Theobromine and Caffeine. M. Malmý. (*J. Pharm. Chim.*, 1921, 23, 89-91.)—A fresh solution of potassium iodobismuthate gives an orange-coloured precipitate both with theobromine and with caffeine; but with old solutions, owing to partial reduction caused by traces of hydrogen iodide, the theobromine coloration changes to chocolate brown. To identify the two alkaloids, 0.05 gm. are mixed with 10 c.c. of water and 0.5 c.c. of fresh potassium iodobismuthate solution, and 5 drops of 10 per cent. iodine, containing about 1 per cent. hydrogen iodide, are then added. In the case of caffeine a bright-red tint is thus produced, whilst with theobromine a brown coloration, changing to chocolate brown in thirty minutes, occurs.

W. J. W.

Proposed Standard for Goldenseal (*Hydrastis*) Root. (U.S. Dept. Agric. Bureau of Chemistry Service and Regulatory Announcements, No. 26, 14.)—As a result of the examination of commercial *Hydrastis* root (in both whole and powdered condition) it is considered that the total ash should not exceed 8 per cent., and that the acid-soluble ash should not exceed 3 per cent.

R. G. P.

Estimation of Hydrastine in *Hydrastis canadensis*. N. Wattiez. (*Chem. Abstracts*, 1921, 15, 145, from *J. Pharm. Belg.*, 1920, 2, 817-819.)—The rhizomes are dried at 50° C., and 7.5 grms. of the powder extracted for three hours with 150 grms. of 70 per cent. alcohol under a reflux condenser. The mixture is then shaken, cooled, and filtered, 120 grms. of the filtrate are evaporated to a syrup, and the berberine precipitated by the addition of sulphuric acid to give a total weight of 15 grms. After standing one hour 1 gm. of talc is added, and the mixture filtered. Ten grms. of the filtrate are made alkaline by the addition of ammonia and shaken frequently during thirty minutes with 40 grms. of ether. After separation, 35 grms. of the ethereal solution are washed with water and extracted with 1 per cent. hydrochloric acid until the aqueous extract yields no precipitate with Mayer's reagent. The extracts are mixed, filtered, warmed to remove ether, cooled, treated with 5 per cent. silicotungstic acid solution, and heated to 100° C. After standing twenty-four hours the mixture is filtered, and the precipitate washed with water until free from acid, and dried to constant weight. The weight of dry residue multiplied by 0.534 gives the amount of hydrastine in 3.5 grms. of the powdered rhizome. For the assay of the extract and tincture of hydrastis by this method, 6 grms. and 30 grms. are taken respectively.

T. J. W.

Alkaloids of Japanese *Corydalis*. Y. Asahina and S. Motigase. (*J. Pharm. Chim.*, 1921, 23, 142.)—The roots of *Corydalis decumbens* (Pers.) contained non-phenolic bases, including protopine, an alkaloid (m.-pt., 142°) perhaps identical with that isolated from *Dicentra formosa* by G. Heyl; and phenolic bases, including bulbocaprine, and another base (m.-pt., 175° C.).

R. G. P.

Lobinol, a Dermatitant from *Rhus diversiloba* (Poison Oak). J. B. McNair. (*J. Amer. Chem. Soc.*, 1921, 43, 159-164.)—Lobinol is the name suggested for the principal dermatitant of the bark of *Rhus diversiloba*. It is an unsaturated

phenolic compound, and possibly contains two hydroxyl groups in ortho-positions. It absorbs bromine freely, and with ferric chloride its alcoholic solution gives a green and then a black coloration, and afterwards a black precipitate or, if the solution be very dilute, a green coloration turned red on addition of sodium carbonate. It forms an acetyl and a benzoyl derivative, an α -naphthylurethane and a phenyl-carbamate. With Millon's reagent it gives a red coloration, and it reduces silver nitrate on heating and ammoniacal silver nitrate in the cold. It hardens at above 96° C. in absence of its enzyme or an oxidising agent, whilst it reacts violently with concentrated nitric acid yielding a nitrate. It dissolves in many organic solvents and in concentrated alkali hydroxide solution, and is precipitated from its alcoholic solution by a number of metallic salts; with barium hydroxide it gives a green precipitate.

T. H. P.

Substitution of the Fruits of *Piper ribesoides* for Cubebs. (U.S. Dept. Agric. Bureau of Chemistry Service and Regulatory Announcements, No. 26, 13-14.)—The fruits of *Piper ribesoides* Wallr. have been substituted for cubebs (*P. cubeba*, L.). True cubebs are 3 to 6 mm. in diameter, with stem-like extensions 5 to 7 mm. in length, and give a crimson-red coloration with concentrated sulphuric acid. The fruits of *P. ribesoides* are 5 to 8 mm. in diameter, with somewhat flattened and curved stem-like extensions up to 13 mm. in length, and give a brownish coloration with sulphuric acid. The stone-cells of the inner layer of the fruit-coat of *P. cubeba* are radially elongated, while those of *P. ribesoides* are generally isodiametric. To conform to pharmacopœial requirements cubebs must not contain more than 5 per cent. of stems or other foreign matter; the Department will recommend the exclusion of cubebs containing over 5 per cent. of foreign matter (including fruits of *P. ribesoides*), except where "definite evidence is furnished that the product will be and is used by a manufacturing firm for a purpose which in no way implies that it is the pharmacopœial article."

R. G. P.

Substitution of Flowers of *Onopordon* Species for Saffron. (U.S. Dept. Agric. Bureau of Chemistry Service and Regulatory Announcements, No. 26, 14.)—The flower-tops (corolla, stigmas, and anthers) of the "cotton" or "Scotch thistle" (*Onopordon* sp.) dyed with tartrazine or commercial Ponceau 2. R., and weighted with borax, potassium nitrate and glycerin have been substituted for saffron (*Crocus sativa* L.). Saffron has a tri-lobed stigma, does not contain anthers or corollas, and imparts a characteristic coloration to water (1 to 100,000, or greater dilution). *Onopordon* has tubular florets (which are much longer than the stigmas of saffron), stamens which show a narrow extension above the anthers, corollas with club-shaped multicellular glandular hairs, and pollen with a peculiar thickened triangular coat. Artificial dyes can be detected by the pink or red coloration with organic solvents, and added salts by the high total ash.

R. G. P.

Examination of Neo-Salvarsan. L. van Itallie. (*Pharm. Weekblad*, 1921, 58, 186-192.)—Neo-salvarsan is an oxidation product of salvarsan, obtained by treatment with sodium formaldehyde sulphonate: $C_{12}H_{11}O_2As_2N_2-CH_2O.SONa$; the com-

mercial substance contains 20 per cent. of arsenic. It may be identified by Denigès-Labot's two reactions : (1) One drop of ferric chloride added to 5 c.c. of neo-salvarsan solution (1 : 1000) gives a red or reddish-violet coloration, which is not destroyed by hydrochloric acid ; (2) 0.5 c.c. of hydrogen peroxide, 0.5 c.c. of ammonia, and 1 drop of 4 per cent. copper sulphate solution give with 5 c.c. of neo-salvarsan solution (1 : 1000) an intense bluish-green coloration, changing to red on addition of hydrochloric acid. Arsenic may be detected by Utz's methods (*Pharm. Zentralhalle*, 1920, 4), but hydrogen peroxide may be substituted for sulphuric acid and ammonium persulphate. The neo-salvarsan (0.05 grm.) is dissolved in 0.5 c.c. of hydrogen peroxide, a crystal of ferrous sulphate added, and the solution is gently heated over a flame till evolution of gas ceases. On adding 3 c.c. of Bettendorf's solution and warming, arsenic separates. Lehmann's method (*Pharm. Zentralhalle*, 1913, 725) may be used for the quantitative estimation.

W. J. W.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Study of Molluscs of the Pacific Coast. P. G. Albrecht. (*J. Biol. Chem.*, 1921, 45, 395-405.)—Five species of molluscs were examined, two of these, *Abalone* and the Pismo clam, being of importance in human dietary. The digestive juices both of the stomach and intestines in each of the species showed an acid reaction and contained a variety of enzymes, although cytase was absent from all. Analyses of the muscle tissues showed a high percentage of ash in all species, whilst the protein content varied from 23 per cent. in *Abalone* to 10.26 per cent. in the Pismo clam. Hydrolysis of the alcoholic extract yielded from 5.26 to 27.2 per cent. of reducing sugars calculated on the dry extract. Creatine and creatinine were identified in *Abalone* muscle, these bases not having been previously recorded in molluscan tissues. Amylase and glycogenase were detected in *Cryptochiton* muscle, and urease in that of the Pismo clam.

T. J. W.

Coffee Bean (*Daubentonia longifolia*) as a Poisonous Plant. C. D. Marsh and A. B. Clawson. (*J. Agric. Research*, 1920, 20, 507-513.)—This plant, sometimes named *Sesbania cavanillesii*, is a shrub belonging to the order *Leguminosæ*, and occurs in the wild state over an area extending from Florida to Texas. In feeding experiments on sheep the smallest dose of the whole plant producing symptoms of poisoning was 0.066 lb. per cwt. of animal, and the minimum fatal dose was 0.11 lb. The seeds are more highly toxic than the leaves and seed pods, although the roasted seeds have been used as a substitute for coffee without deleterious effect. Various closely related plants have from time to time been described as poisonous. The toxic substance in the plant is excreted very slowly by animals, and poisoning may result from the repeated administration of quantities below the toxic dose. The plant is poisonous to sheep, and probably goats, but no data are extant with regard to its effect upon cattle and horses.

T. J. W.

Use of Chloropicrin in the Disinfection of Cereals. A. Piutti and A. Mango. (*Giorn. Chim. Ind. Appl.*, 1920, 2, 677-682.)—Chloropicrin, applied in

the proportion of 20 c.c. per cu. m. of grain store and allowed to act for at least a week at a temperature not lower than 15° C., serves as an efficient parasiticide. It produces no tangible effect on the properties either of the grain or of the flour or bread made therefrom, but it lowers the percentage of germinating corns by about one-third.

T. H. P.

Nutritional Requirements of Yeast. I. Rôle of Vitamins in the Growth of Yeast. E. I. Fulmer, V. E. Nelson, and F. F. Sherwood. (*J. Amer. Chem. Soc.*, 1921, **43**, 186-191.)—The results of the authors' experiments on the stimulation of the growth of yeast by extracts of various materials show that the vitamin water-soluble B is not a necessary constituent of a medium for the growth of yeast, since treatment with alkali does not impair the effects of wheat embryo or alfalfa extracts in this direction. These extracts are found to contain nitrogenous and inorganic materials sufficient to maintain the development of yeast. Any method which utilises the stimulating power of extracts upon the growth of yeast as a quantitative method will yield conflicting results.

T. H. P.

Estimation of Bile Acids. C. L. A. Schmidt and A. E. Dart. (*J. Biol. Chem.*, 1921, **45**, 415-421.)—A mixture of 10 c.c. of bile and 8.5 c.c. of alcohol is heated almost to boiling, filtered, and the residue washed with small quantities of alcohol until the cold filtrate measures 100 c.c. Two 20 c.c. portions are evaporated to dryness in porcelain dishes and the remaining 60 c.c. in a nickel crucible, the residue fused with sodium carbonate and sodium peroxide, and the sulphur estimated as barium sulphate. One of the 20 c.c. residues is dissolved in water, diluted to 10 c.c. and 2 c.c. portions used for the estimation of amino nitrogen by the Van Slyke method (*J. Biol. Chem.*, 1911, **9**, 185). The remaining 20 c.c. of residue are washed into a 10 c.c. flask with 8 per cent. sodium hydroxide, heated to 100° C. for eight hours, cooled, diluted to 10 c.c., and the amino nitrogen estimated in 2 c.c. portions. The difference between the results obtained in the two nitrogen estimations gives the taurine and glycocoll nitrogen of the bile acids. The taurine nitrogen is calculated from the sulphur estimation, and the difference between the amino nitrogen due to bile acids and the taurine nitrogen, less 3 per cent., gives the glycocoll nitrogen. The total maximum error is less than 6 per cent., and the results agree with those of other workers obtained by more lengthy methods.

T. J. W.

Removal of Ammonia from Urine. G. E. Youngberg. (*J. Biol. Chem.*, 1921, **45**, 391-394.) — Five c.c. of urine are diluted to 50 c.c., and 20 to 25 c.c. of the dilute solution are shaken with 3 to 4 grms. of dry permutit for five minutes. After settling, the solution is filtered, and the filtrate, which will then be entirely free from ammonia, may be used for the estimation of urea. Tabulated results given show that the accuracy of the urea estimation is unaffected when the original urine contains excess of ammonia or mineral and urinary salts.

T. J. W.

Estimation of Chlorine in Tissues. R. D. Bell and E. A. Doisy. (*J. Biol. Chem.*, 1921, **45**, 427-435.)—The tissue is heated with about four times its weight of

sulphuric acid, ammonium or potassium persulphate being added at intervals until the solution is colourless. The gaseous products of the reaction pass through absorption tubes containing 20 per cent. sodium hydroxide and 10 per cent. sodium carbonate solutions. On completion of the digestion the absorption tubes are rinsed into a flask, slightly acidified, and gently boiled to remove sulphur dioxide and carbon dioxide, when excess of silver nitrate is added, the solution evaporated and centrifuged. The excess of silver is estimated by titration against potassium iodide, using the Van Slyke-Donleavy method (*J. Biol. Chem.*, 1919, **37**, 551). The results obtained are in good agreement with those obtained by Foster's modification of the McLean and Van Slyke method (*J. Biol. Chem.*, 1917, **31**, 483). T. J. W.

Estimation of Sodium in Blood. E. A. Doisy and R. D. Bell. (*J. Biol. Chem.*, 1921, **45**, 313-323.)—Five c.c. of whole blood or plasma are mixed with 35 c.c. of water and treated with 5 c.c. of 20 per cent. trichloroacetic acid, the whole being diluted to 50 c.c. mixed, and filtered after thirty minutes. To 10 c.c. of the filtrate one drop of concentrated nitric acid is added, and the flask heated over an asbestos sheet until brown fumes are evolved, when the contents are cooled. The precipitating reagent is prepared by dissolving 30 grms. of potassium nitrite (free from sodium) in about 60 c.c. of water, adding 5 c.c. of a 60 per cent. bismuth nitrate solution in 2*N*-nitric acid, dissolving any precipitate in dilute nitric acid, and finally running in a strong solution containing 1.6 grms. of caesium nitrate and 1 c.c. of 2*N*-nitric acid, and diluting the solution to 100 c.c. After standing for twenty-four hours, it is filtered and kept at a low temperature in an atmosphere of coal-gas or hydrogen. The solution containing the sodium is cooled to 10° C. and 3 c.c. of the reagent are added for each mgrm. of sodium expected; coal gas freed from sulphuretted hydrogen is employed to displace the air in the flask, and precipitation is allowed to proceed at 1° C. for twenty-four hours. The precipitate is rapidly filtered through a Gooch crucible, washed with a cold 50 per cent. acetone solution saturated with caesium bismuth nitrate, and finally with pure acetone, after which it is dried at 100° C. to constant weight. The weight of precipitate multiplied by the factor 0.03675 gives the sodium. Since this gravimetric method is liable to certain errors the result may be checked by transferring the precipitate, after drying and weighing, into an excess of standard potassium permanganate solution, adding 10 c.c. of 50 per cent. sulphuric acid, heating the liquid to 75° C., running in excess of standard oxalic acid solution, and finally titrating the excess with standard potassium permanganate solution, when mgrms. of oxygen used multiplied by 0.28738 gives the weight of sodium in mgrms. A further modification is described in which the nitrite present in the precipitate is estimated colorimetrically by the Ilosvay reagent. The results given for the sodium content of blood and urine agree well with those obtained by the usual indirect method, but are less satisfactory when the gravimetric method is adopted.

T. J. W.

Elimination and Fixation of Therapeutic Novarsenicals. Kohn-Abrest, Sicard, and Paraf. (*Comptes rend.*, 1921, **172**, 301-304.)—In the investigation of arsenic in the human body, the presence of about 1 mgrm. of arsenic in the whole

of the viscera may be disregarded. Amounts between 1 and 3 mgrms. indicate therapeutic arsenical treatment, and are usually found in the liver, kidneys, and intestines. The presence in the viscera of 3 to 7 mgrms. is also compatible with novarsenobenzene treatment occurring two to twenty-five days previously, the substance undergoing normal elimination. When, however, the viscera contain mgrms. of arsenic, arsenical poisoning is indicated.

T. H. P.

WATER ANALYSIS.

Occurrence of Hydrogen Sulphide in Waters at Great Depths. O. Luning. (*Zeitsch. Unters. Nahr. Genussm.*, 1921, 41, 29-31.)—Hydrogen sulphide is known to be present in the lower layers of sea and lake waters, the quantity increasing with the depth, although none is found in the surface layers. For instance, in the Black Sea, at a depth of 213 metres the water contains 9.0 mgrms. of hydrogen sulphide per litre, whilst at 2,528 metres the quantity increases to 9.98 mgrms. The author has recently met with an instance where a deep but sluggish river contained large quantities of hydrogen sulphide in its lower layers.

W. P. S.

Estimation of Sulphuric Acid in Water. L. W. Winkler. (*Zeitsch. angew. Chem.*, 1920, 33, 311-312.)—Approximately correct results may be obtained by a rapid nephelometric method of estimating sulphate in water. Five c.c. of the clear water are acidified with 2.5 c.c. of 10 per cent. hydrochloric acid, and treated with 2.5 c.c. of a 10 per cent. solution of barium chloride, and a note taken of the time required for the liquid to become turbid. The amount of sulphuric acid corresponding with the observed time is then obtained by reference to a table or curve, which is preferably constructed from the results given by a standard scale of samples containing known quantities (in mgrms.) of sulphuric acid under the conditions of observation. The results obtained by a gravimetric estimation are inaccurate unless calcium be first precipitated by adding 0.5 to 1.0 gm. of sodium hydroxide and 1 to 2 grms. of sodium carbonate per litre. After the precipitate has subsided, 1 litre of the clear supernatant liquid is acidified with hydrochloric acid and evaporated to dryness, the residue treated with 25 c.c. of water, the solution freed from silica, and the sulphuric acid precipitated from the filtrate.

AGRICULTURAL ANALYSIS.

Investigation into the Occurrence of Different Kinds of Carbonates in Certain Soils. F. Hardy. (*J. Agric. Sci.*, 1921, 11, 1-18.)—Examination of the soils on the north-west coastal belt of the Wash shows that two groups of naturally occurring carbonates may be distinguished: (1) Calcitoid carbonates, being various forms of calcium carbonate easily decomposed by weak acids; (2) Dolomitoid carbonates, comprising magnesite, dolomite, siderite, and other minerals requiring strong acid for their decomposition.

Calcitoid carbonates do not affect the normal rate and process of nitrification, but cultural experiments show that dolomitoid carbonates retard nitrification; ferrous iron is the principal inhibiting factor. For the estimation of the calcitoid

and dolomitoid carbonates the acetic acid method of Linck (*cf.* Johannsen's "Manual of Petrographic Methods," 1918) is not satisfactory, owing to complications arising from the difference in fineness of the dolomite in soils, and other factors. The following method, based on experiments on calcite sand and clay, was adopted: Thirty grms. of air-dried soil are ground to pass a sieve of 0.2 mm. mesh; of two 12-grm. portions one is shaken for forty-five minutes with a rotatory motion with 100 c.c. of CO₂-free water at room temperature, and the other similarly shaken with 100 c.c. of $\frac{N}{4}$ acetic acid. Each is then filtered through dry papers, and the residues washed with CO₂-free water, and dried in an air oven at about 30° C. When dry, the percentage of carbonate in each is determined by the method of Amos (*J. Agric. Sci.*, 1905, 1, 322) on 10 grms. (5 grms. if the carbonate content be high). The figure obtained on the water-treated soil is the percentage of total carbonate, and that on the treated soil (reckoned as CaCO₃) is taken as the dolomitoid carbonate. The difference gives the calcitoid carbonate.

Calcitoid carbonate was present in the soils examined to the extent of about twice the content of dolomitoid carbonate; these soils have not been cultivated; continued cultivation of the soil reduces the quantity of the calcitoid carbonate more than that of the dolomitoid variety, and a fair proportion of total carbonate may be present, yet the soil be lacking in the calcitoid, or most useful variety and sourness result. Soils not derived from marine silts do not usually contain dolomitoid carbonates.

H. E. C.

Studies on Soil Reaction. E. A. Fisher. (*J. Agric. Sci.*, 1921, 11, 19-65).—

I. After reviewing the theories of soil acidity it is shown that the many methods described for its determination yield widely different results, and at best only give empirical information on the quantity of lime required. This is partly due to the past unsatisfactory definitions of soil acidity, which scientifically can only mean the hydrogen ion concentration $[H^+]$ in the soil solution, and which is most conveniently expressed as $-\log [H^+]$ (the reciprocal of $\log [H^+]$). The older titrimetric methods fail because of adsorptive and "buffer" effects which are bound up with the solid phase, so that the reaction of a soil solution is only constant when it is in contact with the solid. Much improvement is, however, effected by the use of the sulphonephthalein dyes instead of litmus. A critical study of the Hutchinson-MacLennan method (*Chem. News*, 1914, 10, 61, abstr. ANALYST, 1914, 450, and *J. Agric. Sci.*, 1914, 6, 323) shows that the results are from three to twenty times as great as those of the colorimetric method, the higher values occurring with slightly acid soils. This is due to adsorption by soil particles, which is expressed by the equation $y/m = kC^{\frac{1}{p}}$, where "y" is the lime absorbed by "m" grms. of soil, "C" the amount of lime in the liquid phase, and "k" and "p" are constants. Study of this equation shows that the lime requirement, which cannot in practice be differentiated from the acidity, as shown by this method, will vary with the size of the particles; the fact that the soil does not adsorb equivalent quantities of different bases is also a source of error. Further it shows that the lime requirement of the soil is the sum of two quantities—(1) the lime required to neutralise soil acids, and (2) the lime actually

adsorbed by the soil complex. It is at present impossible to differentiate between these; different classes of soils give very different adsorption curves on plotting the above equation, so that results are unreliable; they can, however, be rendered comparable by interpolating the results from an experimental curve of $\log y/m$ against $\log C$.

II. Methods of estimating $[H^+]$ or $-\log [H^+]$ are electrometric or colorimetric, the latter really depending on standardisation by the former. The colorimetric method is based on the availability of a number of indicators with a wide range of colour changes from $-\log [H^+] = 1$ to $-\log [H^+] = 10$. *Preparation of the Solutions required.*—Standard Buffer Solutions. (*cf.* Clark and Lubs, *J. Bact.*, 1917, 1, 109-191; *J. Biol. Chem.*, 1916, 25, 479).—M/5 HCl, M/5 KH_2PO_4 , M/5 acid potassium phthalate, M/5 boric acid, M/5 KCl and M/5 NaOH, are all made with conductivity water. Potassium acid phosphate, phthalate, and chloride are recrystallised from conductivity water, and dried at 110° to 120° C.; boric acid is recrystallised and air-dried below 50° C.; sodium hydroxide is purified by a special method, and standardised against the acid potassium phthalate. The following mixtures are made: (A) Phthalate mixtures, giving $-\log [H^+] = 2.2$ to 3.8 ; (B) Phthalate-NaOH mixtures giving $-\log [H^+] = 4.0$ to 6.2 ; (C) KH_2PO_4 -NaOH mixtures giving $-\log [H^+] = 5.8$ to 8.0 , and (D) Boric acid-KCl-NaOH mixtures giving $-\log [H^+] = 7.8$ to 9.8 . These solutions keep for about six weeks if a little calomel be added. The following indicators are used (*cf.* Clark and Lubs, *loc. cit.*) for the ranges of $-\log [H^+]$ named: Thymol-blue, 1.2 to 2.8 ; brom-phenol-blue, 3.0 to 4.6 ; methyl-red, 4.4 to 6.0 ; brom-cresol-purple, 5.2 to 6.8 ; brom-thymol-blue, 6.0 to 7.6 ; phenol-red, 6.8 to 8.4 ; thymol-blue, 8.0 to 9.6 . These are used in the form of 1.2 per cent. aqueous solutions of the sodium salts (0.6 per cent. for phenol-red, and 0.1 grm. of methyl-red in 500 c.c. of diluted alcohol). On account of dichromatism the determinations are made in daylight. *Method of Using Colour Standards.*—To 10 c.c. of standard buffer solution are added, in three test tubes $4''$ by $5/8''$, four drops of indicator (methyl-red, six drops) and the tube closed with a rubber cap. These are compared with 10 c.c. of the solution the $-\log [H^+]$ value of which is required, by a modification of Walpole's method (*Biochem. J.*, 1910, 207). In the case of soil the solution is prepared as follows: 50 grms. of soil (3 mm. sample) are shaken with 100 c.c. of conductivity water in an end-over-end shaker for half an hour; after allowing the mixture to stand for a few minutes the supernatant liquor is centrifuged at 3,000 revs. per minute for ten minutes, and the colour with the indicator measured as above. Experiment shows that filtration disturbs the results. To determine the lime requirement colorimetrically, varying amounts of $\frac{N}{8}$ barium hydroxide solution are added to mixtures of soil with twice its weight of water and the $-\log [H^+]$ value measured as before; the results are plotted and the amount required to make $-\log [H^+] = 7.07$ interpolated and calculated to CaO. The results are not absolute values, but are comparable from sample to sample. The fineness affects the values of $-\log [H^+]$ obtained; 100 mesh samples give lower results than 3 mm. samples, but the centrifuged liquor is turbid and requires flocculation by addition of calcium acetate, which decreases the observed acidity.

H. E. C.

The Preparation and Fractionation of Humic Acid. V. A. Beckley. (*J. Agric. Sci.*, 1921, 11, 66-68.)—From results of attempted determinations of the methoxy content of carefully prepared humic acid, it is concluded that the humic acid of Ehrenberg and Bahr (*J. für Landw.*, 1913, 61, 427) and Oden, which they considered to be homogeneous and of basicity between three and four, is really a mixture of a tribasic and a tetrabasic acid. The humic acid, which is insoluble in ether and other solvents, is divisible into two parts, both acidic, one soluble and the other insoluble in pyridine. The alcohol-precipitated humus may therefore be further fractionated into Hoppe-Seyler's hymatomelanic acid, which yields melanin and other compounds, and into acids which by pyridine extraction give a soluble and an insoluble humic acid.
H. E. C.

The Formation of Humus. V. A. Beckley. (*J. Agric. Sci.*, 1921, 11, 69-77.)—The author confirms Fenton's observation that humus is formed artificially by condensation of ω -hydroxymethyl-furfural. At ordinary temperatures the change takes place very slowly, but on boiling a 12 per cent. hydrochloric acid solution of hydroxymethyl-furfural, humus is rapidly produced together with some furfural. Cane sugar, when boiled with 3 per cent. hydrochloric acid, produces hydroxymethyl-furfural, the colour changing through yellow, red, brown, to black with the production of humus. Experiments with amino-acids and polypeptides also confirm the finding of Maillard that humus results, and it is shown that hydroxymethyl-furfural is the intermediate product. It is probable that the furfural produced from hexoses and hexosans by boiling with hydrochloric acid results from the decomposition of the hydroxymethyl-furfural, and it is unnecessary to postulate the formation of furfural to account for the production of humus. Experiments on rotted straw and on soil confirm the suggestion that humus is produced naturally from carbohydrates by the above changes, and although hydroxymethyl-furfural is unstable and polymerises rapidly to humus, evidence of its presence has been obtained. There is no evidence of the formation of hydroxymethyl-furfural during the decomposition of cellulose by *Spirochaeta cytophaga*.
H. E. C.

ORGANIC ANALYSIS.

Estimation of Organic Compounds by Oxidation with Chromic Acid. H. Cordebard. (*Ann. Chim. analyt.*, 1921, 3, 40-53.)—Oxidation is carried out by treating the substance with an excess (20 c.c.) of *N*-dichromate solution, adding 20 c.c. of sulphuric acid, and gently boiling the mixture for five minutes at a temperature not above 160° C. After cooling, the liquid is diluted with 100 to 150 c.c. of water and a slight excess of $\frac{N}{10}$ ferrous ammonium sulphate is added and titrated back with $\frac{N}{10}$ dichromate solution. The results obtained with a large number of compounds of different types show that the amount of oxygen required under these conditions is in accordance with theory. Preliminary experiments have also led to the following conclusions: (1) Oxidation takes place normally in the case of substances containing C_2H_5 groups or several CH_3 groups attached to one carbon atom, except where there are groupings in which two atoms of carbon and four atoms of hydrogen only require 2.1 atoms of oxygen for their conversion to acetic acid.

(2) From compounds containing nitrogen united with oxygen nitrogen is evolved, whilst compounds containing amino groups evolve ammonia. (3) In the case of compounds containing metalloids, the latter are converted into the anhydrides of maximum oxygen content. (4) In the case of compounds containing halogens, the latter are liberated and, acting as oxidising agents, spoil the determination. R. G. P.

Estimation of Arsenic in Organic Compounds. G. R. Robertson. (*J. Amer. Chem. Soc.*, 1921, **43**, 182-185.)—The following method, occupying some two hours, may replace the tedious Carius process. About 0.2 gm. of the organic arsenic compound is treated in a 150 c.c. pyrex Erlenmeyer flask with 5.5 c.c. of concentrated sulphuric acid, and when it is completely wetted or dissolved, 1 c.c. of fuming nitric acid is added and the flask set on a hot plate under a hood; the plate is heated so that water in a similar flask boils actively but not excessively, the actual temperature of the mixture undergoing oxidation being approximately 250° C. In about thirty minutes arsphenamine or any of its intermediates are likely to be oxidised completely, whilst such refractory substances as tolylarsenic acid are partly oxidised and partly charred. In general it is safe after an hour to cool the flask slightly, add to it 10 to 15 drops of fuming nitric acid, and heat for five minutes, by which time the oxidation is complete. After 1 gm. of solid ammonium sulphate has been cautiously added, the vessel is shaken for a few moments until the nitrogen has escaped, allowed to cool to below 100° C., and then cooled under the tap, the clear, colourless mixture being carefully diluted with water to a volume of 60 to 70 c.c. One gm. of potassium iodide and a few scraps of porous tile are added, and the flask, fitted with a simple bulb trap, such as an inverted 25 c.c. flask with a side vent, heated on the same hot plate as before until the volume of the liquid is reduced to 40 c.c. The bulb trap is rinsed into the flask, 0.01*N*-thiosulphate solution is added dropwise until the colour of iodine disappears, and the liquid instantly diluted with cold water to 100–120 c.c. and transferred to a flask containing 50 c.c. of 4*N*-sodium carbonate solution. The residual acid is then slightly over-neutralised with sodium bicarbonate, starch solution being added, and the arsenite titrated with standard iodine solution. If a high degree of accuracy be desired, the sodium bicarbonate may be replaced by a buffer solution of phosphates, which keeps the solution exactly neutral during titration. T. H. P.

Absorption of Carbon Monoxide by Cuprous Ammonium Carbonate Solutions. W. R. Hainsworth and E. Y. Titus. (*J. Amer. Chem. Soc.*, 1921, **43**, 1-11.)—The capacity of a cuprous ammonium carbonate solution of given cuprous copper content to absorb carbon monoxide depends on the free ammonia content of the solution and on the partial pressure of the carbon monoxide above it, as well as on the concentration of the cuprous copper. The absorption takes place in consequence of the formation in solution of an unstable compound probably containing one mol. of carbon monoxide per gm.-atom of cuprous copper. Cupric copper is reduced to cuprous copper fairly rapidly by carbon monoxide, but the reduction of cuprous copper to metal is much slower. The presence of oxygen in small proportions in gaseous mixtures from which carbon monoxide is to be removed by absorption will prevent the precipitation of copper and increase the absorptive capacity of the

solution. Practically complete removal of carbon monoxide from used cuprous ammonium carbonate solutions may be effected by heating to 75° C. at ordinary pressure in an inert atmosphere, the absorptive capacity being restored unimpaired.

T. H. P.

Estimation of Small Proportions of Carbon Monoxide in Air and in Flue Gases. D. Florentin and H. Vandenberghe (*Comptes rend.*, 1921, 172, 391-393.)—Two methods for estimating carbon monoxide have been investigated: (1) *The Iodine Pentoxide Method* (cf. Teague, *ANALYST*, 1920, 45, 459).—This method is not specific, since the oxide is reduced by many other gases difficult, if not impossible, to remove. The results obtained show that the commercial iodine pentoxide should be purified by solution in water, slow evaporation on a sand-bath at 150° C., and heating in a current of air at 200 to 210° C. to expel occluded iodine and decompose any iodine tetroxide. The proportion of carbon monoxide in the gas should not exceed 0.1 per cent., since otherwise the iodine may be deposited on the cold parts of the apparatus in fine spangles removable only by prolonged washing. The apparatus should contain no glass wool or asbestos, which fix small proportions of iodine, and there should be no rubber connection between the iodine pentoxide tube and the iodine-absorption vessel. The reaction between carbon monoxide and iodine pentoxide being slow, a U-tube containing a 20 cm. column of the powdered pentoxide at 140° to 150° C. should be used, and the volume of gas passed through it should not exceed 700 to 800 c.c. per hour. (2) *The Blood Method*.—The air is freed from oxygen by shaking it with a concentrated sodium hydroxide solution of sodium hydrosulphite, and is then passed through a 1 per cent. blood or hæmoglobin solution at the rate of about 600 c.c. per hour. The number, N , of c.c. of oxygen-free air required to cause the appearance of the absorption bands of the hæmoglobin-carbon monoxide compound in the spectrum of the blood solution varies with the number of parts, M , of carbon monoxide in 100,000 parts of the original air in accordance with the following table, which it is advisable for the observer to check:

N	90	125	150	200	300	400	500	750	1000	1300	2200
M	100	60.6	45.0	25.0	13.3	8.8	6.5	3.7	3.1	2.0	1.0

If the gas to be analysed contain more than 0.1 per cent. of carbon monoxide, it should be diluted before testing. For the gas from the combustion of different fuels, such as sawdust, lignite, etc., this method gives absolutely constant results, whereas the iodine pentoxide method mostly gives quite erroneous results owing to the presence of various organic substances.

T. H. P.

Application of the Law of Hydrolysis to the Determination of Molecular Weights. H. Colin and A. Chaudun (*Comptes rend.*, 1921, 172, 278-280.)—The course of the enzymic inversion of sucrose is governed by the relation between the concentrations of sugar and enzyme. With a given quantity, n_1 , of the catalyst, measured by a certain volume of an enzyme solution, there corresponds a definite weight, a_1 , of sucrose. If n_1 c.c. of the enzyme act on a quantity of sucrose not greater than a_1 , the velocity of hydrolysis diminishes throughout the action in accordance with Wilhelmy's law, but if the weight of sugar exceed a_1 , the velocity

remains constant until the excess of sugar has disappeared, and subsequently diminishes until the action is complete. Similar relations are met with if the sucrose be replaced by raffinose, gentianose, etc., from which the levulose residue is detached by invertase. It is, indeed, found that the weights of the different sugars capable of fixing one and the same amount of sucrase are in the ratios of the molecular weights of the sugars. When solutions of various concentrations of sucrose, raffinose, and gentianose were hydrolysed under similar conditions as regards temperature and content of sucrase—the sugar being always in excess with reference to the enzyme—the weights of the three sugars present in the solutions when the velocity of hydrolysis began to decrease were respectively 1.83, 3.18, and 2.69 grms., which are proportional to the molecular weights of the three sugars. T. H. P.

Methylation of Cellulose. III.—Homogeneity of Product and Limit of Methylation. W. S. Denham. (*J. Chem. Soc.*, 1921, 119, 77-81.)—By repeated treatment of cellulose with sodium hydroxide and methyl sulphate a product containing 44.6 per cent. of methoxyl has been obtained, which indicates that the limit of methylation corresponds to the formation of trimethyl-cellulose. The solubility of methylated cellulose in Schweitzer's reagent decreases as the methoxyl content increases; products containing 40 per cent. of methoxyl are nearly homogeneous, and are practically insoluble in Schweitzer's reagent and in alcohol, acetone, or chloroform. Fehling's solution is not reduced by the highly methylated products.

H. E. C.

Volumetric Estimation of Mixtures of Acids and of Bases, and of Polybasic Acids or Bases. H. T. Tizard and A. R. Boeree. (*J. Chem. Soc.*, 1921, 119, 132-142.)—The conditions for the accurate titration of mixtures of two weak acids, or bases, present in the ratio of 1 to x equivalents, or for the titration of dibasic acids or bases to the half-way point, are that K_1/xK_2 should be at least 2.5×10^4 , and that an indicator should be available which changes over at $C_H = \sqrt{xK_1K_2}$ (where K_1 and K_2 are the dissociation constants). If K_1/xK_2 be at least 2.5×10^4 an accuracy of 1 per cent. is possible, and if this ratio be 10^6 , an accuracy of 0.1 per cent. is possible. Experiments and the theory show that the following mixtures can be accurately titrated: Ammonia and aniline using cresol-red; ammonia and β -picoline using methyl-orange; phenol and acetic acid using azolitmin or cresol-red; trichloroacetic and acetic acids using methyl-violet. Monochloroacetic acid cannot be titrated in the presence of acetic acid, since K_1/K_2 is too small; similarly, succinic acid cannot be titrated to the half-way point, although maleic acid can be if methyl-orange be used. If the salt of a polybasic acid having very different dissociation constants be used as a hydron regulator, a slight excess will have a large effect; hence an appropriate mixture of ammonia and acetic acid is preferred for this purpose.

H. E. C.

Iodoform Reaction. N. Schoorl. (*Pharm. Weekblad*, 1921, 58, 209-210.)—In Kunz's reaction (*ANALYST*, 1920, 45, 458), ammonium persulphate may be used instead of the potassium salt. In the former case the iodoform has an intense

orange colour and is devoid of crystalline structure. The reaction becomes extremely sensitive if acetone be used, and is effective with concentrations of 1 : 100,000. In addition, with acetone instead of alcohol, the reaction may be carried out with 2 *N*-sodium carbonate instead of sodium hydroxide solution.

W. J. W.

Estimation of Oil of Turpentine. H. Salvaterra. (*Chem. Zeit.*, 1921, 45, 133-135, 150-151, 158-159.)—Three suitable methods for the estimation of oil of turpentine are described. In a modification of Tausch's method, 20 c.c. of the sample are introduced into a 2-litre flask containing 300 c.c. of water and 20 c.c. of 80 per cent. acetic acid. Fifty-one grms. of powdered mercuric oxide are slowly added, and then 10 c.c. of methyl alcohol, after which the flask is connected with a reflux condenser and heated on a water-bath for two to three hours. The undecomposed hydrocarbons, present as impurities, are then distilled off in a current of steam and measured. For the examination of oil of turpentine adulterated with benzine 20 c.c. are added to 20 grms. of potassium bromide and 10 c.c. of water. Fifty c.c. of 50 per cent. hydrogen bromide are run in slowly with continuous cooling, after which the temperature is allowed to rise to 40° to 50° C. After standing for one hour, the solution is neutralised, and the bromine is fixed by addition of solid sodium carbonate and powdered arsenic acid until the solution becomes almost colourless. It is then distilled in steam. If solvent naphtha be present, excess of bromine must be avoided. Twenty c.c. of turpentine oil are treated with 12 grms. of sodium bromate and 10 c.c. of water. Hydrogen bromide is then added till the coloration persists for five minutes, the operation being preferably performed with exclusion of daylight. The temperature is then raised to, and maintained at, 20° to 25° C. for half an hour, and the solution is neutralised and distilled in steam as before. When pine oil (*Kienöl*) is present as the adulterant the bromine oxidation may be incomplete, and it is advisable to add 2 c.c. of hydrogen bromide after the coloration has lasted for ten minutes. The method is unsuitable in case of adulteration with tetrahydronaphthalene. In the iodine method 10 c.c. of turpentine oil are introduced into a flask containing 40 grms. of iodide, 52 grms. of mercuric chloride, and 100 c.c. of alcohol, the mixture being kept cool and then allowed to stand in the dark for six hours. A concentrated solution of potassium iodide is then added, and excess of sodium thio-sulphate solution, after which the mixture is neutralised with sodium hydroxide, and finally distilled in steam. By applying both the mercuric acetate method and either the bromine or iodine process, it is possible to estimate the amount of pine oil in the adulterants, as by the first method 10 per cent. of this substance separates in the distillate, which is not the case with either of the other two processes. The difference in the respective separations, multiplied by 10, will give the content of pine oil.

W. J. W.

Estimation of Aromatic Hydrocarbons in Mixtures with Petrol. H. T. Tizard and A. G. Marshall. (*J. Soc. Chem. Ind.*, 1921, 40, 20-25T.)—The method described is developed from the one previously worked out by Thole (*ANALYST*, 1919, 44, 144.)—Ten c.c. of the mixture are added to 10 c.c. of freshly distilled aniline in a

large test tube fitted with a stirrer and a thermometer reading to 0.1° C. The mixture is heated until a clear solution is obtained, when the temperature is allowed to fall slowly until the mixture suddenly becomes turbid, at which point the thermometer is read. This temperature is called the "aniline point." A second portion of the mixed hydrocarbons is shaken with three times its vol. of 98 per cent. sulphuric acid for half an hour, the residue being separated, washed with sodium hydroxide solution and water and dried over calcium chloride, and the "aniline point" again determined. The difference in degrees between the two results represents approximately the percentage by weight of aromatic hydrocarbons in the original mixture, whether consisting of benzene, toluene, or xylene, or mixtures of the three. In the case of mixtures containing two or more aromatic hydrocarbons, the percentage of each may be determined by fractional distillation of the original mixture, and determining the respective "aniline points" of the fractions distilling up to 95° C., from 95° to 122° C., and from 122° to 150° C. Assuming the first fraction to contain all the benzene, the second the whole of the toluene, and the third all the xylene from the original mixture, satisfactory results were obtained with known mixtures. The accuracy of the results may be increased by a determination of the "aniline point" for small fractions obtained close to 95° and 122° C. by a preliminary distillation and plotting a curve of the "aniline points" and mean boiling points of the fractions, when the maxima indicate the most correct "cutting points" for the distillation of the mixture under examination. T. J. W.

Lamp Method of Estimating Sulphur in Petroleum Spirits. J. S. Jackson and A. W. Richardson. (*J. Inst. Petrol. Technol.*, 1921, 7, 23-34.)—In the lamp described, the ordinary wick is replaced by a bundle of capillaries which are spread by means of a central air tube lightly jammed into position. By this arrangement the air does not pass through the spirit to be burnt, so that fractionation is greatly reduced, if not eliminated. A mixing chamber is provided above the tops of the capillaries, and the luminosity and size of the flame are easily controlled by the air supply. Any sulphur held on the capillaries can be washed off, oxidised, and included in the estimation, but the quantity of sulphur thus held is usually negligible. W. P. S.

Laboratory Testing of Oil Shale for Oil and Ammonia Yield. E. L. Lomax and F. G. P. Remfrey. (*J. Inst. Petrol. Technol.*, 1921, 7, 34-47.)—*Oil*.—This is estimated by distillation with steam. The retort used consists of a 20-inch length of 3-inch iron pipe, mounted horizontally and fitted at each end with reducing sockets; the inlet end is connected with a steam supply and the outlet end with a condenser, vessel for receiving the distilled oil, a second condenser, vessels containing oil (these act as gas scrubbers), and an aspirator filled with water (to measure the gas yield). One kilo of the shale is placed in the retort, which is then heated, and steam at 180° C. is admitted slowly; the heat is increased gradually to ensure a steady distillation of oil, and at the end of the operation the retort is heated to dull redness. The crude oil is further examined as to its sp. gr. solidifying point, sulphur content, water content, and fractional distillation. The first fraction, light naphtha,

volatile below 150° C., is distilled directly; steam is then introduced and fractions collected between 150° and 200° C. (heavy naphtha), 200° and 250° C. (light kerosene), 250° and 300° C. (heavy kerosene); the higher boiling oils are then collected in fractions of 5 or 10 per cent. each. The naphtha fractions are refractionated, and the high boiling oils are examined as to their sp. gr., paraffin wax content, etc. *Ammonia*.—The retort described above is used, the shale being placed in the middle section of the retort, and the two ends filled with ignited broken fire-brick. The ends of the retort are brought to a dull red heat before the shale is heated; steam at 180° C. is then admitted, and the whole retort heated rapidly to a dull redness. The receiver and absorption vessels contain dilute sulphuric acid, and at the end of the operation their contents are mixed, diluted to a definite volume, and the ammonia estimated in the usual way. W. P. S.

New Specific Test for Phenol. G. Rodillon. (*J. Pharm. Chim.*, 1921, **23**, 136-137.)—A drop of sodium nitrite solution (10 per cent.) is mixed with about 10 c.c. of the liquid in a test-tube, and a few c.c. of sulphuric acid are added without mixing; in the presence of phenol a ring, which is emerald-green below and ruby-red above, forms at the zone of contact. It is necessary that the phenol should be free from substances producing colorations with sulphuric acid. R. G. P.

INORGANIC ANALYSIS.

Properties of Selenium Oxychloride. V. Lenher. (*J. Amer. Chem. Soc.*, 1921, **43**, 29-35.)—Selenium oxychloride (m.-pt. 8.5°, b.-pt. 176.4° at 726 mm., n_D^{20} 1.6516, specific electrical conductivity about 9.6×10^{-4} at 28° C.) is pale yellow and transparent to ordinary light, but it makes a practically perfect screen for all wavelengths below 4050. The oxychloride dissolves sulphur, selenium, tellurium, bromine, iodine, and phosphorus, the last violently; boron, silicon, and carbon in its various forms are unattacked in the cold. Most metals react with the oxychloride, giving the chloride of the metal and selenium monochloride. A solution of sulphur trioxide forms a powerful solvent which dissolves the oxides of aluminium, chromium, the rare earth metals, titanium, columbium, molybdenum, vanadium, and uranium, that of tantalum slightly, but not those of zirconium and tungsten. Strontium carbonate dissolves easily and barium carbonate slightly, calcium carbonate being insoluble; in the cold zinc carbonate dissolves at once, but the carbonates of lead, copper, nickel, and cobalt are only slightly attacked, whilst that of magnesium becomes colloidal. Saturated paraffin hydrocarbons are attacked slowly by the oxychloride at a high temperature, whilst unsaturated aliphatic hydrocarbons, as well as turpentine, etc., unite directly and often violently, and aromatic hydrocarbons form mixtures capable of physical resolution; thus such separations as heptane from amylene or from benzene may be effected. Protein-containing materials, such as hair, bristles, silk, and leather, are dissolved in the cold, but cellulose is not attacked. Pure and vulcanised rubber, vulcanite, bakelite and its allies, gums, resins, dried paints, shellac, dried varnish, lacquer, agar-agar, celluloid, gelatin, and glue dissolve in the oxychloride, as do the natural asphalts, resins, and bitumens when they are unsaturated.

T. H. P.

Use of Gallium Ferrocyanide in Analysis. L. E. Porter and P. E. Browning. (*J. Amer. Chem. Soc.*, 1921, **43**, 111-114.)—When separated as ferrocyanide gallium forms a gelatinous precipitate difficult to filter. It is found that if a mat of fine filter-paper fibre, best made by scratching some paper with a knife, be washed into the funnel containing a double filter-paper, the gallium ferrocyanide is retained provided that the suction used be not too strong; the first portion of the filtrate may have to be returned to the filter. Direct ignition of the ferrocyanide and subsequent weighing of the mixed gallium and ferric oxides yield high results. If, however, the gallium ferrocyanide be treated with an alkali hydroxide solution and carbon dioxide passed through the liquid to saturation, the gallium is precipitated quantitatively as hydroxide or basic carbonate in a form easy to filter and to wash free from ferrocyanide; the precipitate is ignited and weighed as Ga_2O_3 . Another method which works well and is rather quicker than the carbon dioxide method consists in oxidising the gallium ferrocyanide to ferricyanide by hydrogen peroxide in alkaline solution and boiling the solution with ammonium chloride, gallium hydroxide free from cyanide radicles being thus precipitated. This procedure cannot be followed if zinc be present, owing to the insolubility of zinc ferricyanide in acid and in ammonium hydroxide. In this case, either (1) the mixed ferrocyanides are dissolved in sodium hydroxide solution, the bases precipitated together by means of carbon dioxide, and the gallium separated from the zinc by dissolving the precipitate in hydrochloric acid and boiling the solution with acid ammonium sulphite to precipitate the gallium; or (2) the alkaline solution of the ferrocyanides is treated with hydrogen sulphide to precipitate the zinc, the zinc sulphide filtered off, and the gallium recovered from the filtrate free from ferrocyanide either by precipitation with carbon dioxide or by boiling with ammonium chloride after oxidation of the excess of sulphide and of the ferrocyanide by means of hydrogen peroxide.

T. H. P.

Separation of Gallium from Indium and Zinc by Fractional Crystallisation of the Cæsium Gallium Alum. P. E. Browning and L. E. Porter. (*J. Amer. Chem. Soc.*, 1921, **43**, 126.)—The separation of gallium and indium by the fractional crystallisation of the cæsium sulphate alums of these elements (*cf.* Uhler and Browning, *Amer. J. Sci.*, 1916, **42**, 389) has been further studied to determine quantitatively the progress of the separation. A mixture containing 26.5 parts of gallium oxide to 73.5 parts of indium oxide was converted into the cæsium alums; fractional crystallisation of the latter yielding successive fractions containing respectively 100.0, 95.3, 64.3, 23.4, 1.9, and 0.6 (mother liquor) per cent. of gallium oxide in the total oxide present. Gallium may be freed from zinc in a similar manner.

T. H. P.

Qualitative Separation and Detection of Uranium, Vanadium, and Chromium when Present together. P. E. Browning. (*J. Amer. Chem. Soc.*, 1921, **43**, 114-115.)—The author modifies the method given by Noyes (*J. Amer. Chem. Soc.*, 1908, **30**, 481) for separating uranium, vanadium, and chromium, the introduction and subsequent removal of lead being avoided. The solution containing

chromic and vanadic acids, together with a uranium salt, is made faintly alkaline with ammonia and then faintly acid with acetic acid, and treated with ammonium phosphate. The uranium phosphate thus precipitated is filtered off and washed, the filtration being facilitated, if necessary, by addition of shredded filter-paper. The precipitate is treated on the filter-paper with acidified potassium ferrocyanide solution, a characteristic reddish-brown stain resulting if uranium be present. The filtrate is treated with a distinct excess of sulphurous acid, which reduces the chromic and vanadic acids, the solution being successively boiled to remove the excess of sulphurous acid, treated with distinct excess of bromine water, and boiled to expel the excess of bromine. The bromine oxidises the vanadium to the acid condition, but does not affect the reduced chromium. While the excess of bromine is being boiled away some of the chromium is precipitated as phosphate, and this precipitation may be rendered complete by adding ammonia in slight excess and boiling off the excess; a green precipitate indicates chromium. After removal of the chromium by filtration the filtrate is made alkaline with ammonia and saturated with hydrogen sulphide, which gives the pink or violet oxysulphide of vanadium if the latter be present.

T. H. P.

Volumetric Determination of Iron by Means of Titanous Salts. W. M. Thornton, jnr., and J. E. Chapman. (*J. Amer. Chem. Soc.*, 1921, **43**, 91-102.)—The conditions under which Knecht and Hibbert's method (*Ber.*, 1903, **36**, 1549) may be employed have been examined. The replacement of the bead valve of the original apparatus by a three-way cock is recommended. Titanous sulphate was used instead of the chloride, since it gives a solution which appears to maintain its strength indefinitely. Although many objections have been raised against ferrous ammonium sulphate as an oxidimetric standard, it is found that this salt, if of good quality, serves as a sufficiently accurate standard for titanous solutions, since only the total iron content is of importance in this case. Where there is no special objection, 10 c.c. of 10 per cent. ammonium thiocyanate solution is a safe and ample quantity of indicator, and in no case should there be present less than 2.5 c.c. of such solution. The influence of certain acids and of other interfering substances, the preliminary oxidation for the determination of total iron, the direct determination of ferric iron, the verification of the iron determinations, and the determination of iron and available oxygen in artificial manganese dioxide, are among the other questions considered.

T. H. P.

Reaction of Manganese Salts. V. Maeri. (*Ann. Chim. analyt.*, 1921, **3**, 55-56.)—On adding a few drops of a solution of an alkali oxalate and of acetic acid to an alkaline solution of a manganese salt, a rose-red coloration is produced. The reaction is more sensitive if the solution of manganese salt be boiled before the addition of oxalate and acetic acid, and does not occur in presence of strong mineral acid. A definite coloration is obtained with one drop of a 1 : 1000 manganese solution. Phosphoric acid appears to possess the same action as oxalic acid.

R. G. P.

Interference of Phosphates with Detection of Manganese. T. Sabalitschka and H. Niesemann. (*Ber. Deut. Pharm. Ges.*, 1921, **31**, 30-36.)—The method given by Schmidt ("Anleitung zur qualitativen Analyse") for the detection of

manganese fails in presence of a large proportion of barium or strontium phosphate, but the following modification yields satisfactory results: The precipitate formed by ammonium sulphide is washed with water containing ammonium sulphide and dissolved in cold 5 per cent. hydrochloric acid solution, the solution being filtered and a small portion of the filtrate tested for iron and phosphoric acid. If the latter be found, the acid solution is evaporated with 2-3 grms. of concentrated sulphuric acid, 2-3 grms. of saturated potassium sulphate solution, and, if iron be present, a little nitric acid, until dense white fumes are evolved. The mixture of sulphates thus obtained is treated with alcohol, the undissolved residue being taken up in a little boiling dilute hydrochloric acid, and filtered from the insoluble sulphates of barium, strontium, and calcium. The filtrate is treated with ammonia solution, and the precipitated aluminium, iron, and chromium hydroxides removed by filtration. To the filtrate are added 3-5 grms. of ammonium chloride and also ammonium sulphide, the precipitated manganese and zinc sulphides being dissolved on the filter in a little dilute hydrochloric acid and the solution thus obtained heated to expel hydrogen sulphide, treated with excess of sodium hydroxide solution, and tested for manganese and tin. The filtrate from the sulphide precipitate, which may contain calcium and magnesium, is evaporated to dryness, the residue being heated to expel ammonium salts, moistened with hydrochloric acid, and dissolved in water; the filtered solution is treated with ammonia solution in excess, and tested for calcium and magnesium. The above procedure admits of the detection of 0.05 gm. of zinc oxide and 0.05 gm. of crystallised manganese chloride in the mixture. In presence of smaller amounts of zinc and manganese, the amounts of these metals in the alcoholic extract must also be considered, the process then requiring some further modification.

T. H. P.

Iodimetric Titration of Chromium. J. M. Kolthoff. (*Zeitsch. anal. Chem.*, 1920, 59, 401-415.)—Zulkowsky's method gives good results if the acid concentration be not below 20 per cent. 4 N; and direct titration may be carried out. The concentration of the iodine and dichromate is of small importance. Presence of molybdate retards reaction between iodine and chromium, and further promotes by-reactions between the chromium and thiosulphate, especially in sunlight. Ferrous salts exert a positive catalytic effect in weak acid solution, but with strong acid solutions the interaction of the iodine and chromium salt is impeded. The reaction velocity is not affected by temperature. Even dilute dichromate solutions may be satisfactorily titrated; dilution of the solution after adding the reagents is unnecessary.

W. J. W.

Acidimetry of Boric Acid. J. A. M. v. Liempt. (*Rec. Trav. Chim. Pays-Bas*, 1920, 39, 358-370.)—The author investigates electrometrically the method based on the addition of polyhydric alcohol to the solution of boric acid previous to titration. The hydrogen ion concentration is plotted against the volume of alkali added, in presence of various quantities of mannitol, glycerol, or lævulose, and if sufficient of the alcohol be present the curves show a marked inflection at the neutralisation point. Curves for mixtures of acetic and boric acids and for borax are included.

O. E. M.

Solid Lubricants.* (*Dept. Scientific and Ind. Research. Bull. No. 4, 1920, pp. 28.*)—A description is given of the characteristics of solid materials used for lubricating purposes, of the action of solid lubricants, their analysis, methods of application and drawbacks, and of the results obtained by their use in practice. The following analyses by L. Archbutt show the composition of various commercial lubricating graphites calculated on the dry material free from volatile matter :

	Acheson Graphite. No. 1,340.	Graphite from "Aquadag."	Graphite from "Oildag."	"Foliac." No. 100.	"Graphite." B. 1,371.	"Foliac." No. 2.	"Foliac." No. 1.	"Foliac." No. 101.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Graphite	99.34	98.68	97.58	99.62	93.74	98.06	90.06	99.95
Mineral matter (ash) ...	0.66	1.32	2.42	0.38	6.26	1.94	9.94	0.05

The "aquadag" contained 15.06 per cent. of material insoluble in water after coagulation by slight acidification, and the "oildag" containing 11.2 per cent. of graphite, etc., insoluble in ether. In the Acheson graphites the principal impurities were iron and alumina, whilst in the "foliac" natural graphites silica (1.60 to 4.40 per cent. of the ash) was the main impurity. "Foliac" graphite No. 100 was nearly chemically pure, the flakes appearing absolutely black when not reflecting light.

PHYSICAL METHODS, APPARATUS, ETC.

Use of Phosphoric Acid instead of Sulphuric Acid in Melting-Point Determinations. A. Graustein. (*J. Amer. Chem. Soc.*, 1921, 43, 212-213.)—Phosphoric acid, prepared by heating orthophosphoric acid to the highest temperature desired, in order to obtain the necessary concentration, is recommended in place of sulphuric acid in melting-point baths. A vessel of resistance glass, such as pyrex, must be used. Clouding of the acid is removed by adding water and reheating, and discoloration by introduction of a few crystals of sodium nitrite. T. H. P.

Temperature as a Function of the Absolute Viscosity of Oils and other Liquids. H. Schwedhelm. (*Chem. Zeit.*, 1921, 45, 41-42.)—The absolute viscosity of an oil, calculated from the Engler viscosity, is related to the temperature as shown by the equation: $\frac{z}{G} = \left(\frac{z'}{G}\right) (H^{t-t'})$, in which z and z' are absolute viscosity at temperatures t and t' , whilst G and H are constants, a table of which is given for a number of oils. In the case of mineral lubricating oils, G varies from 0.01887 to 0.02857, and H from 1.01485 to 1.01858; with rape oil, $G=0.01887$ and $H=1.01218$; with water, $G=0.00145$ and $H=1.01278$; and with alcohol, $G=0.000160$ and $H=1.00441$. The equation permits values to be calculated where experimental determination—at high or low temperatures—is difficult. O. E. M.

* By permission of H.M. Stationery Office.

Fractional Precipitation. P. Jolibois, R. Bossuet, and Chevy. (*Comptes rend.*, 1921, 172, 373-375.)—The apparatus previously designed for the systematic study of consecutive precipitation in a chemical reaction (*ANALYST*, 1920, 45, 65) is now applied to the investigation of fractional precipitation. Use is made of two dilute solutions, *A* and *B*, which give a precipitate when mixed, the titre of *A* being constant, whilst that of *B* is varied in different experiments and so calculated that a certain known fraction of the substance is precipitated. The compositions of the liquid and the precipitate indicate the fractionation effected. By this means it is shown that addition of sodium hydroxide solution to a mixture of equal volumes of $\frac{N}{50}$ cupric nitrate and silver nitrate results in the complete precipitation of the copper before any silver is precipitated. With a mixture of $\frac{N}{50}$ solutions of nickel and cobalt chlorides, however, the fractionation effected by precipitation with sodium hydroxide solution is imperfect. The precipitate is always richer in nickel than would be the case if the two metals were equally precipitated, and the content of cobalt in the supernatant liquid increases with the content of sodium hydroxide in the precipitating solution. It is probable that basic salts are formed during this precipitation, since the quantities of metal remaining in the liquid decrease almost proportionally to the quantity of sodium hydroxide introduced.

T. H. P.

Conductimetric Titration. A. Heldring. (*Chem. Weekblad*, 1921, 18, 96-97.)—The conductimetric estimation of organic acids, involving decomposition of their salts in suspension in a mixture of alcohol and water by means of strong acids, may be simplified in various ways. Stirring of the liquid in the thermostat may be more conveniently effected by compressed air than by mechanical means; the air may be previously heated if desired. Removal of the electrolyte vessel and electrodes for the purpose of shaking by hand after addition of acid is also obviated by employing air agitation; to avoid loss of alcohol, it is advisable to pass the air first through a mixture of alcohol and water. Several estimations may be carried out in the same thermostat by the use of connectors. To secure more rapid equilibrium in the alcoholic liquid by increasing the temperature in the thermostat, it is necessary to substitute higher alcohols, also miscible with water, for ethyl alcohol.

W. J. W.

Critical Temperature of Solution and Turbidity-Temperature of Hydrocarbons, etc. N. Chercheffsky. (*Ann. Chim. analyt.*, 1921, 3, 53-55.)—The following solvents are recommended for use in the determination of the critical temperature of solution: (a) For petroleum spirit, paraffin lamp oil, lubricating oil, vaseline, ceresine and paraffin waxes—ethyl alcohol, 96.5 per cent.; (b) for turpentine oils—ethyl alcohol, 90 per cent.; and (c) for castor oil—ethyl alcohol, 85 per cent. And for the determination of turbidity temperature: (a) For petroleum spirit and paraffin lamp oils—acetic anhydride; (b) for lubricating oils—equal volumes of glacial acetic acid and toluene; (c) for vaseline—amyl alcohol, 129° to 131°; (d) ceresine and paraffin waxes—crystallisable benzene; and (e) for oleines and unsaponifiable matter—aniline and glacial acetic acid.

R. G. P.

An Improved Pyknometer. R. Saar. (*Chem. Zeit.*, 1921, 45, 102-104.)

—To simplify calculation, the pyknometer, which is only suitable for liquids of specific gravity approximating to unity, has a narrow neck with divisions corresponding to mgrms. of water, 150 above and 150 below the mark indicating the nominal content. The instrument being charged and its temperature brought to 15° C., the level and weight of the liquid are ascertained. The number of mgrms. above the normal mark is subtracted from, or the number below added to, the net weight of the liquid.

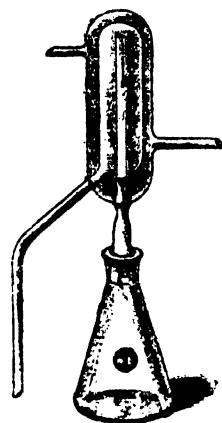
O. E. M.

Soxhlet Apparatus. L. Soep. (*Chem. Weekblad*, 1921, 18, 97.)—The lateral vapour tube enters the upper portion of a Soxhlet apparatus near the base, and is then continued upwards in the form of a spiral surrounding the extraction thimble. By this arrangement the solvent vapours serve to heat the condensed liquid.

W. J. W.

Distillation Apparatus for Small Quantities. C. Woytacek. (*Chem. Zeit.*, 1921, 45, 82.)—The apparatus consists of a 100 c.c. conical distilling flask with ground-in condenser of the form shown.

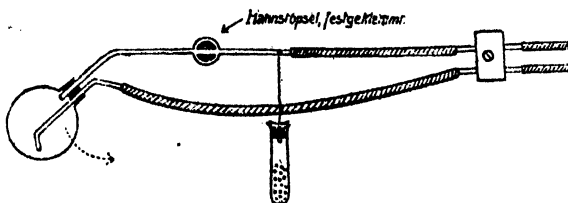
O. E. M.



Glass Autoclave. Schmidt. (*Chem. Zeit.*, 1921, 45, 131.)—For reactions in which appreciable evolution of gas takes place, or in which the introduction of gas under pressure is desired, a glass autoclave may be employed. The apparatus includes two condensers, and is provided with an inlet tube for the gas and an outlet tube connected with a safety-valve. It may be employed with pressures up to 4 atmospheres.

W. J. W.

Non-return Arrangement for Water Pumps. H. Dorsch. (*Chem. Zeit.*, 1921, 45, 32.)—A narrow-bore glass cock is connected at one end by a length of pressure tubing with the apparatus to be exhausted, and at the other end carries a small flask. The arrangement is free to rotate around the plug of the cock, held in a clamp, as fulcrum. By means of pressure-tubing the flask is connected with the pump, and receives any water flowing back from the same, the weight of which depresses it until the cock is shut. When the action of the pump improves, the water is drawn off, the flask is raised by a counterweight, and the cock is opened.



O. E. M.

Improved Soxhlet Apparatus. O. Hagen. (*Chem. Zeit.*, 1921, 45, 19.)—To facilitate removal of samples during an extraction the tube connecting the boiling-flask and the apparatus is bent into a ring which is provided with a draw-off cock at its highest point. A funnel directs the condensed solvent into the centre of the extraction thimble.
O. E. M.

Repair of Crystallised Glass Apparatus. A. F. O. Germann. (*J. Amer. Chem. Soc.*, 1921, 43, 11-14.)—In the devitrification of glass the silica which separates forms an infusible coating over the surface of the glass, and may under certain conditions dissolve in the underlying silicate, so that the surface clears up, whilst under other conditions it yields calcium silicate, fusing with difficulty; in which case well-defined crystal surfaces may appear. According to the older hypothesis, this phenomenon is due to slow volatilisation of sodium oxide from glass near its softening temperature, and a common practice has been to reintroduce the lost alkali; this is, however, always difficult. It is found that the tendency to devitrify may be removed by rinsing the parts of the glass to be fused with dilute hydrofluoric acid; this treatment renders possible the repair of old apparatus.
T. H. P.



REVIEWS.

A SYSTEM OF PHYSICAL CHEMISTRY. Vol. II.: THERMODYNAMICS. Third Edition
By W. C. McC. LEWIS, M.A. (R.U.I.), D.Sc. London: Longmans, Green
and Co. Price 15s. net.

Professor Lewis's work is sufficiently well known as a standard textbook of physical chemistry to require but little description. The third edition of Volume II., in which the facts of physical chemistry are treated from the standpoint of thermodynamics, contains a few additions in the light of recent work, notably an extensive new section in Chapter VIII., dealing with the advances made by the American school of physical chemistry on solutions of electrolytes and ionic activity. In Chapter XI. the section on Donnan's Theory of Membrane Equilibria is enlarged, and in the same chapter is an entirely new section, giving a comprehensive account of the Micelle Theory of Colloidal Electrolytes. In the treatment of both of these subjects considerable space has been given to experimental details; such descriptions form a relieving break in a volume, the pages of which are, of necessity, mainly mathematical.

Chapter VII. also contains some additions dealing with advances made in the field of electrochemistry. A recapitulation of the contents of a few of the chapters serves to give an idea of the scope of the volume. Chapter X. contains a thorough account of the theoretical aspect of the Phase Rule, and gives a number of well-chosen illustrative examples of the use of this law. Chapter XII. deals with

Chemical Affinity; Chapter XIII. is devoted to the Nernst Heat Theorem and its bearing on chemical phenomena. The first part of Chapter XIV. describes the theory of Light Radiation, and the second and larger part is a systematic account of Photochemistry.

As the author states in the Preface to Volume I., first edition, the book is not intended for beginners. With the volume at present under review, a working knowledge of the facts and elementary theory of physical chemistry is assumed. The student need not, however, possess any previous knowledge of thermodynamics, as the first four chapters are chiefly devoted to a treatment of thermodynamics in general. The exhaustive manner in which the thermodynamical theorems used are deduced from first principles makes these chapters extremely valuable, not to the chemist only, but also to the student of pure physics.

It is rare that a rigorous and methodical system of treatment of a science is found together with lucidity and ease of style. That Professor Lewis has succeeded in this difficult task may be seen in the popularity of the first two editions of his work, which may, indeed, be said to be indispensable to all students taking an Honours Degree in chemistry, or pursuing the study of physical chemistry.

H. R. AMBLER.

OPTICAL METHODS IN CONTROL AND RESEARCH LABORATORIES. London: Adam Hilger, Ltd. Pp. 30. Price 1s. 6d. net.

This little work draws attention in concise language to the applications of optical determinations to applied chemistry; it is divided into four sections, dealing with the spectrometer and spectrograph for emission spectra, absorption spectra and spectrophotometry, the refractometer, and the polarimeter respectively. That an optical determination is rapid, that it involves the loss of very minute quantities of a sample, and that the information deduced from it is often of much value, are commonplaces, and there are few analytical laboratories without a spectroscope, a refractometer, and a polarimeter; it is safe, however, to say that even the most persistent devotee of optical methods will find much that is suggestive and new.

It is marvellous what a wide range is touched upon in so small a book; references to a long list of papers giving the details of working methods are very numerous. The one omission is the failure to emphasise the importance of the information to be obtained by combining an optical with another physical determination; thus refractive index plus density will give control figures for both extract and alcohol in many substances, will estimate malt solids and cod-liver oil in malt and oil preparations; refractive index and rotatory power will enable the crystallisation of alkaloid solutions to be followed, and the combined determinations often yield results unobtainable by either method alone.

The fact that the book is published by a leading firm of optical instrument makers suggests that it is an advertising brochure, but its useful suggestiveness is so great that it is a form of advertisement to be encouraged.

H. DROOP-RICHMOND.

LABORATORY MANUAL OF ORGANIC CHEMISTRY. By H. L. FISHER. London: Chapman and Hall. 1920. Price 19s. 6d. net.

This book should prove a useful guide to the study of practical organic chemistry; the material is well arranged and presented in a manner calculated to maintain the interest of the student.

Amongst the more notable features is the account of experiments dealing with the conversion of pinene to camphor, in which the student actually carries out a synthesis involving a fairly lengthy series of operations. It is sometimes difficult to get the youthful chemist to realise the importance of obtaining his products, not only in a state of purity but also in good yield, and the performing of a synthesis, involving a number of separate reactions, is perhaps one of the best means of emphasising the importance of the second desideratum.

The completeness and excellence of the section on the determination of carbon, hydrogen, and nitrogen gives one a feeling of regret that the author does not include an account of methods for the estimation of halogens, sulphur, and phosphorus and also of the commoner organic radicals.

The questions at the end of the various sections of the book are generally well chosen, and should go far towards arousing an intelligent interest in the subject, and so tend to eliminate what might be called "cookery book" chemistry.

The volume is well produced; the clearness and excellence of the diagrams leave little to be desired, and the typographical errors are relatively few in number. Attention may be called, however, to an obvious error of punctuation on p. 264, and to the use of the word "practice" on p. 117 where the verb is intended, although possibly this may be intentional. The following minor points may also be noted: The misspelling of bromnaphthalene on p. 17; the density of aniline is quoted on p. 161 at $1\frac{1}{8}^{\circ}$ —presumably meaning the density of aniline at 16° compared with that of water at 4° —the more usual nomenclature $1\frac{1}{8}^{\circ}$ seems to be preferable; on pp. 178 and 204 the boiling-points ascribed to phenol do not agree—this is the more regrettable as the author lays much stress on the "correction" of thermometric data.

In the preparation of a tertiary alcohol by means of Grignard's reaction (p. 70) it is surely not necessary to have so large an excess of ethyl bromide as that given in the directions—namely, 100 per cent. In the experience of the reviewer a 10 to 20 per cent. excess of ethyl bromide is usually quite sufficient to bring about complete solution of the magnesium.

There are several innovations in terminology—namely, the use of 1° , 2° , 3° for primary, secondary, and tertiary, and of the word "nitronation" for the process of nitration as distinct from the formation of nitric esters. The wisdom of the first appears to be questionable, and any gain of accuracy from the use of the second would probably be outweighed by the confusion it would cause.

JOSEPH KENYON.

RECENT ADVANCES IN ORGANIC CHEMISTRY. By ALFRED W. STEWART, D.Sc. Fourth Edition. London: Longmans, Green and Co. 1920. Price 21s. net.

The appearance of a fourth edition of this well-known book so soon after that of the third is in itself a clear indication of the value and interest of the work. The present volume does not differ greatly from its predecessors in general character; hence it is unnecessary here to outline its scope. New matter has been added to bring each section thoroughly up to date, so that those who wish to keep themselves abreast of the times may have every confidence that no really important work, dealing with the theoretical side of organic chemistry, has been overlooked. It is obviously impossible in some 350 pages to go deeply into detail; but there are abundant references to original papers of dates extending to 1920.

Of the new material incorporated in the present edition, special attention may be called to the fresh section added to the chapter on the polypeptides. Here is traced the connection between these synthetic substances and the naturally occurring proteins. The lines along which the compositions of the latter are being approached are clearly indicated, but little hope is given that this most complex problem will be solved in the immediate future. One of the most interesting and important additions in the present volume is the last chapter, entitled "Some Unsolved Problems." Why is carbon unique in its capacity for chain and ring formations? Why should the *number* of carbon atoms in a ring have so great an influence? What are the principles underlying "Crum Brown's Rule"? Why are some compounds stable, while others of similar structure are either unstable or even non-existent? What is the mechanism of intramolecular change? These are a few of the problems presented to the reader as awaiting solution. In some cases indications are given of possible solutions; in others we must look to future years to provide the key. None the less the statement of these difficulties is of great value. In the first place it reminds us of the limitations of our present knowledge: how we are still very much on the outer surface of things, as it were, and really know little of internal structure and of the forces at work in holding atoms in combination. Useful, nay indispensable, as our ordinary plane formulæ are, we need to be reminded that they can hardly portray the actual arrangement of atoms in molecules; yet three-dimension formulæ have at present little application apart from the phenomenon of stereoisomerism. Recent research has thrown much light on the structure of crystals and of atoms; is it too much to hope that in the near future some new method of investigation will reveal to us the secrets of molecular structure? Secondly, to be confronted with an array of problems awaiting solution is not only a stimulus to further research, but also an indication of the topics on which fresh knowledge is specially required. As the author points out in his preface, too much time is spent, one feels inclined to say wasted, on the preparation of numberless new compounds of scant interest in themselves, which, at present at any rate, have no practical application, and also are unlikely to add anything to our understanding of the essential nature of chemical combination. This book provides a clear exposition of the methods by which, in the last few years, the compositions of many complex

substances have been unravelled ; it cannot fail to be of great assistance to those who will be called upon to handle the still more complex problems of the future.

The two indexes are fully adequate to their purpose. A. F. KITCHING.

NUCLEIC ACIDS: THEIR CHEMICAL PROPERTIES AND PHYSIOLOGICAL CONDUCT. By WALTER JONES, Ph.D., Professor of Physiological Chemistry in the Johns Hopkins Medical School. London. Second Edition. New York: Longmans, Green and Co. 1920. Price 9s. net.

Progress in all fields of biochemical research is so rapid that it is difficult for the literature to keep pace with it. Having regard to the very great importance and to the great complexity of the problems with which biochemists are concerned, it is perhaps more necessary than in any other branch of chemical science that the investigator and the student alike should have the opportunity from time to time of obtaining, by reference to authoritative and reliable monographs, a clear survey of the existing state of knowledge. It is for this reason especially that we welcome the second edition of Dr. Jones' monograph, the first edition of which appeared in 1914. Since that date considerable additions have been made to our knowledge of the nucleic acids and their physiological behaviour, and these are all fully and clearly dealt with in the present volume.

A detailed review of this work in a journal devoted to analytical chemistry would clearly be out of place. As showing, however, how important a bearing biochemical investigations may have upon purely biological questions, the reviewer cannot refrain from referring to the very interesting and recently established fact that in respect of purine metabolism, the higher apes resemble man in that they are unable to convert uric acid into allantoin, whilst in the lower mammals, including the monkeys, this change is readily effected.

To many analytical chemists, and especially to those who have to deal with problems of food chemistry, some knowledge of the chemistry and physiological behaviour of the breaking-down products of the nucleo-proteins is necessary, and no safer and clearer guide than the work under review could be recommended or, indeed, could be desired.

In a very comprehensive Appendix will be found collected the more important analytical reactions of the purine and pyrimidine derivatives, as well as of the nucleosides and nucleotides—information which, as far as the writer knows, is not available in a collected form in any other publication.

The book is written not merely in a lucid, but in a very attractive manner, and by one who can claim to speak on the subject with authority. It is remarkably free from typographical and other mistakes, and is well printed.

The reference on page 15 (in connection with the structural formula of uric acid) to Medicus having "made a place for himself in history by his ability to guess the correct formula" is not very generous, even though it contains a grain of truth. In his Paper, published in 1875, dealing with the constitution of uric acid and of some of its derivatives, Medicus based his conclusions on a consideration of

the experimental results obtained by Strecker and others, and arrived at the formula which was subsequently proved by Emil Fischer and by Behrend and Roosen to be the correct one. It may be mentioned, incidentally, that it was this same work of Strecker which led directly, in the hands of Horbaczewski, to the synthesis of uric acid.

This book is deserving of unqualified commendation, and constitutes a very valuable addition to the excellent series of monographs on biochemistry, for the editing of which Dr. Plimmer and Dr. Gowland Hopkins are jointly responsible.

A. CHASTON CHAPMAN.

AN INTRODUCTION TO THE CHEMISTRY OF PLANT PRODUCTS. By PAUL HAAS, D.Sc., and T. G. HILL, A.R.C.S. Vol. I. Third Edition. London: Longmans, Green and Co. 1921. Price 16s. net.

The fact that in bringing out the third edition of this book the authors have found it necessary to enlarge it so as to occupy two volumes is sufficient proof of its popularity. The present Vol. I. contains most that was in its predecessors, some improvements having been introduced in the arrangement and order of the subject-matter. The section devoted to colloid chemistry is practically new and now presents a clear and interesting summary of the present position of that subject. The second volume, which is in preparation, is to be devoted more exclusively to plant physiology.

The authors have, for the purpose of compiling this manual, laid a very large field of literature under contribution, and as a compendium of chemical information of use to botanists we do not think that it has a rival in this language. If the book has a fault, it is that the student is given too little guidance in selecting the fundamental and important from among a mass of facts and conflicting opinions. For example, the section on starch (p. 114 *et seq.*) forms an instance where clearness has been sacrificed to multiplicity of detail.

The practical part of the book consists of a number of qualitative experiments grouped at the end of each section, illustrating some of the reactions of the substances previously dealt with theoretically, together with a selection of quantitative estimations of the same substances. It is in the latter that we think that the authors have missed an opportunity for affording help to the botanist with biochemical leanings. In the section on "fats," for example, the routine methods for fat analysis employed in commercial laboratories—saponification, acid, acetyl, and iodine values, etc.—are described, but no attempt is made to show how these may be applied to tracing the changes which take place in the fat of plant tissues during various stages of growth.

The interpretation of the insoluble bromide test (Hehner and Mitchell) is misleading; the weight of the precipitate obtained is *not* proportional to the amount of unsaturated fatty acids present, but is merely an indication of the amount of triglycerides containing acids of the linolenic series. The method selected for the preparation of lecithin by means of the cadmium chloride compound might usefully have been exchanged for the more recent acetone-precipitation method.

The section on carbohydrates gives a useful account of the most important members of the group, though a mistake in describing xylose as optically inactive has crept in. The section on the estimation of sugars fails to appreciate the difficulties of the botanist in the preliminary treatment of tissue. Examples of the estimation of mixtures of sugars are given, but the *combined* use of the polarimeter and the reduction method is not described. The estimation of maltose is made needlessly confusing, and the hydrolysis of this substance is termed "inversion." It is difficult to understand why the reduction methods of Fehling and Pavy are described when they have been superseded by many others equally simple and more reliable, such for example as that of Bertrand.

The chapter on plant pigments is a clear account based largely on the work of Willstätter and his pupils, and includes some discussion on the physiological aspect of the subject. It is surprising to find that the authors have included (p. 256) a method for the preparation of phycoerithrin from red sea-weed, consisting in the precipitation of the aqueous extract of the tissue with methylated spirit, by which means the pigment (together with protein and other impurities) is thrown down. "The pigment in a pasty mass . . . is dried in a vacuum," after which it is found to give most of the protein reactions. "From these and other facts it is concluded that phycoerythrin is a colloidal nitrogenous substance allied to the proteins." It is from methods and conclusions such as this that a knowledge of biochemistry should protect the botanist.

The chapter on enzymes is a clear treatment of the broad theoretical aspects of the subject, besides giving much detailed information of use to botanists. It is a little misleading to head a paragraph "The Isolation of Zymase" when the preparation of an aqueous extract of yeast containing the enzyme is intended.

M. STEPHENSON.

THE YEASTS. By ALEXANDRE GUILLIERMOND; translated and thoroughly revised in collaboration with the original author by FRED WILBUR TANNER. Pp. 409. New York: John Wiley and Sons. 1920. Price 33s. net.

This English edition is based on Guilliermond's "Les Levures," which was published in 1912, and to which numerous additions and modifications have now been made by both author and translator.

The language of the book is often slipshod and sometimes not readily intelligible, and orthographic and other errors, more especially in the names of authors, are to be numbered by the dozen. The following few examples must suffice: "Separated closely from" (p. 7); "The cells bud generally at their extremities; this is exceptional only when lateral budding is proven" (p. 10); "S. Ludwigii did not keep longer than 2 years, often 6, . . ." (p. 165); "concurrence" is used to denote "competition" (p. 154); "microscopic" appears for "macroscopic" (p. 167); "Sur l'habitat et la conversation des levures spontanées" (p. 256). Schutzemberger, DuClaux, Linder, Boyen-Jensen, Noegeli, Pearse, and Van Tieghm all occur, some of them repeatedly, while Haydruck is given for Hayduck throughout.

Luckily chemistry and physics do not figure to any considerable extent in the

book, for in these subjects the author—or is it the translator?—rapidly gets out of his depth. Thus we find: "They [certain yeasts] act on the melibiose which they change to dextrose and galactose; this is fermented to *d*-glucose" (p. 63); "hydrocarbon" is used twice on p. 54 and again on p. 161 with the meaning of "carbohydrate," whilst on p. 75, "hydrocarbon compounds" serves as a heading for substances able to supply the requirements of yeasts as regards carbon compounds; the "thermal deathpoint" is defined as "the amount of heat which is necessary to destroy the yeast" (p. 168).

In spite of these and many similar blemishes, the book well deserves perusal by all interested in yeasts, whether from a scientific or from an industrial point of view, and it is to be deplored that the publishers have found it necessary to demand such a high price. The treatment of the subject is predominantly botanical and in this direction may be regarded as reasonably complete, although one or two striking omissions force themselves on the notice of the reader. Noteworthy among these is the absence from the section dealing with the characteristics of alcoholic fermentation, and also from the excellent bibliographical index, of any reference to Adrian Brown's work on the influence of oxygen and concentration on alcoholic fermentation, on the specific character of the fermentative functions of yeast cells, etc.

The subjects dealt with in the various chapters are: Morphology and development of the yeasts; cytology of yeast; physiology, nutrition, respiration, and alcoholic fermentation; origin of the yeasts, their position in classifications of the fungi and their systematic relationships; methods of culture and isolation of yeasts; methods for characterising and identifying yeasts; variation of species; classification of the yeasts; family of saccharomycetes; family of non-saccharomycetes or doubtful yeasts; pathogenic yeasts; fungi related to the yeasts. In general the facts are presented fairly and lucidly, and objection can scarcely be taken to the prominence given, here and there, to the author's own views and results. Especially valuable are the last four chapters, which contain descriptions of about 300 yeasts and allied micro-organisms, and occupy nearly 200 out of the 380 pages of the text of the book; this section is well illustrated.

The bibliographical index extends to nearly 30 pages, and is followed by name and subject indexes. The printing and general style of the book are of the high standard of excellence usually associated with Messrs. John Wiley's productions.

T. H. POPE.

BIBLIOTHECA CHEMICO-MATHEMATICA. By H. ZEITLINGER and H. C. SOTHERAN.
Vols. I. and II. Pp. 964, including Index. London: H. Sotheran and Co.
Price 63s. net.

One of the authors points out in his preface that this is "perhaps the first historical catalogue of science published in any country, at least as giving at once the current price of each book included, bibliographical particulars, and many historical references, both in the descriptions themselves and in the notes." Although the price of each book is quoted, it must not be assumed that this work is merely a

booksellers' catalogue, as it really contains a great deal of useful and interesting information which is not readily accessible elsewhere.

One is, perhaps, tempted to enquire why this work includes some comparatively modern books, and was not restricted to those published before a certain date; presumably this was impossible in what is in part a price-list, but the effect is curious, and it is surprising to find such works as Newton's "Principia" of 1686 rubbing shoulders with the second edition of Lewkowitsch's "Oils and Fats of 1898." Unfortunately the catalogue is not complete, and while a good many of the earlier books dealing with analysis are included no reference is made, for example, to Winter Blyth's "Foods," though more recent and less important works are mentioned. In spite of its incompleteness, this catalogue cannot fail to be of permanent value as a book of reference, particularly to those who are interested in the fascinating study of the history of chemical science.

A rather striking point is the low market value of many of the older and rarer books, even such a landmark in the progress of science as a first edition of Newton's "Principia," which is quoted as "excessively rare," may be had for a modest eighteen guineas. When one considers the high prices often paid for first editions of "belles lettres," one feels that there must be a lack of appreciation of historical science, or that those interested in it are too few or too impecunious to cause the price of old and rare scientific books to rise to a high level.

The catalogue is very well arranged and indexed, and contains numerous reproductions from photographs of interesting plates. The authors are to be congratulated on the result of their painstaking and laborious task.

R. G. PELLY.



THE INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

THE FORTY-THIRD ANNUAL GENERAL MEETING OF THE INSTITUTE OF CHEMISTRY was held on March 1 at the Institute's house in Russell Square. Sir Herbert Jackson, the retiring President, in the course of his address, remarked that Government departments and official authorities generally had shown more inclination in recent times than in the past to accord higher recognition to the services of men of science. The conditions of appointments of chemists, both in the Government service and in industry, had been improved, and until the close of 1920 the profession had been in almost a unique position in that very few indeed of its members were disengaged. The prevailing depression in industry had had some effect on the employment of chemists, but even now less than fifty were disengaged out of nearly 3,300 on the Roll. The By-laws of the Institute had undergone revision to provide for the representation of chemists from various parts of the country and practising in different departments of work, and alterations have been made to define more specifically certain points relating to the ethics of the profession and further to restrict the membership of the Institute to British subjects. The Institute was taking part in many matters affecting the public life of the country where chemistry was concerned, and the Annual Report showed that chartered professional bodies of this character were able to render the State valuable service. The greater consideration given to science by the Government was an encouragement to the coming generation of chemists to follow a career of essential and vital importance to the needs of the country. It would probably be regarded as desirable at the present moment for the Council of the Institute, without taking part in politics, to give expression to their views on the grave importance of maintaining in this country industries on which not only the future development of our chemical industry and many allied industries depends, but the future of a very large number of students of chemistry who are now in the course of training. The Institute was entrusted by its Charter with securing the supply of well-trained chemists, but, unless a great chemical industry were maintained, there would be a very poor prospect for them. The President maintained that the Institute had throughout its existence fulfilled the purposes for which it was incorporated. It had kept alive the corporate spirit of the profession, the means whereby the opinions and views of its members found expression in matters of public concern. It was the officially recognised public body to whom the country could turn for advice and guidance on matters relating to the profession. It rendered every possible assistance to those who intended to follow the profession of chemistry, and promoted and maintained the highest standard of training and competence for that profession. It registered the trained and competent, and thereby supplied competent professional service for the country. Moreover, it promoted the strictest integrity on the part of its members in their dealings with one another and with the public, and fostered by every means in its power the status of the profession. He was confident that it would continue to gain strength and prestige.

The adoption of the Report and a vote of thanks to the President for his address were moved by Sir William Tilden and unanimously carried.

The Officers and Council were elected as nominated, and the Censors and Auditors were elected for the ensuing year. Mr. Alfred Chaston Chapman, F.R.S., succeeds Sir Herbert Jackson as President.

SCIENTIFIC AND INDUSTRIAL RESEARCH DEPARTMENT.

THE Secretary announces that the following Associations have been approved by the Department as complying with the conditions laid down in the Government scheme for the encouragement of industrial research :

The British Research Association for Liquid Fuels. *Secretary*, Percy Still, M.I.E.E., 19, Cadogan Gardens, S.W. 1.

The Research Association for Cast Iron and Allied Industries. *Secretary*, T. Vickers, 174, Corporation Street, Birmingham.

The Secretary also announces that the Lord President of the Council has established an Inter-Departmental Committee on Patents, with the following terms of reference: (1) To consider the methods of dealing with inventions made by workers aided or maintained from public funds, whether such workers be engaged (a) as research workers, or (b) in some other technical capacity, so as to give a fair reward to the inventor and thus encourage further effort, to secure the utilisation in industry of suitable inventions, and to protect the national interest; and (2) to outline a course of procedure in respect of inventions arising out of State aided or supported work, which shall further these aims and be suitable for adoption by all Government Departments concerned.

The Secretary to the Committee is Mr. A. Abbott, to whom all communications should be addressed at 16 and 18, Old Queen Street, Westminster, London, S.W. 1.

**THE FINE CHEMICALS INDUSTRY.**

THE leading British makers of fine chemicals have published a letter in which they point out that this industry is of national importance, and desire to submit to public knowledge the following facts: (1) The application of chemical science to industry is essential to the preservation of our world position in commerce; (2) essential plant erected during the war years could be adapted to peace purposes, but must become idle and useless unless the State take measures for its safeguarding; (3) the fine chemical industry provided during the war, *inter alia*—(a) essential medicinal preparations; (b) higher forms of explosives and gases; (c) photographic chemicals, and (d) research chemicals; (4) British science and chemical manufactures are in no respect inferior to the science and manufactures of competing nations.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

ORDINARY MEETING, APRIL 6, 1921.

HELD at the Chemical Society's Rooms, Burlington House. Mr. Alfred Smetham, President, in the Chair.

Certificates were read for the first time in favour of Messrs. William Ellard Woolcott and Thomas Henry Pope, B.Sc., F.I.C. Certificates were read for the second time in favour of Messrs. Percy N. Mould and W. J. Wright, F.I.C.

The following were elected Members of the Society: Messrs. Jules Cofman-Nicoresti, Walter K. Fletcher, William Singleton, James Darnell Granger, Ph.D. (Berlin), F.I.C., Edward Bradford Maxted, Ph.D. (Berlin), B.Sc. (Lond.), Russell George Pelly, F.I.C., and Francis George Henry Tate.

The following papers were read: "The Estimation of Strychnine in Scale Preparations containing Quinine and other Cinchona Alkaloids," by T. F. Harvey, F.I.C., and S. Back; "The Action of Water on Lead," by J. C. Thresh, D.Sc., M.D., F.I.C.; "A Colour Reaction for Aconite," by S. Mallanah, M.D., D.P.H.; and "A Method for the Determination of the Acidity of Coloured Solutions," by J. L. Lizius, B.Sc., A.I.C.



OBITUARY NOTICES.

CHARLES ALEXANDER CAMERON.

ALTHOUGH Sir Charles Cameron was personally known to but a small proportion of our present members, his name has been so long and so closely connected with the Society that his death, which took place on February 27, will be felt as a loss by all of us.

Cameron, who was born in 1830, was the son of an officer who served in the Peninsular War and in the war against the United States. He was educated in Dublin and in Guernsey, where his parents lived from 1844 to 1846. On the death of his father he returned to Dublin, and entered the laboratory of Messrs. Bewley and Evans, apothecaries. Here he acquired a practical knowledge of pharmaceutical chemistry, meanwhile attending lectures on chemistry, physics, and medicine at the Royal Dublin Society.

In 1852 the Dublin Chemical Society was founded, and Cameron was elected its Professor of Chemistry ; and this appointment soon led to the Professorship of Chemistry and Natural Philosophy in the Dublin School of Medicine, to which he was appointed in 1856. Soon afterwards he became Professor in the Royal College of Surgeons, Ireland.

In 1860 the first Food and Drugs Act was passed, but, as the appointment was made permissive, few authorities troubled to appoint Public Analysts, and, in consequence, the Act soon became a dead letter. Dublin, however, was one of the few towns to make use of the Act, and, in 1862, Cameron was elected Public Analyst for that city—a post which he retained until his death. He was the third Public Analyst in the United Kingdom to be appointed under the Act. Subsequently, when the Act of 1870 was passed, rendering the appointment of Public Analyst obligatory, Cameron was chosen by no fewer than thirty-three counties and boroughs as their official analyst. In fact, from the number of these appointments he was often humorously referred to as “The Public Analyst for Ireland.”

In the early days of our Society, Cameron contributed numerous papers on the analysis of milk, water, and drugs, which were published in *THE ANALYST*. He served on the Council in 1878-81, became Vice-President in 1882-3 and again in 1889-90, and held the office of President in 1893 and 1894. He was also a Vice-President of the Institute of Chemistry. In addition to his chemical posts, he held numerous medical appointments, being Medical Officer as well as Public Analyst for Dublin, and he served as President of the Royal College of Surgeons of Ireland.

He was an honorary M.D. of the Royal and National Universities of Ireland and an honorary Fellow of the Royal College of Physicians of Ireland. In 1885 he was knighted in recognition of his efforts to improve the dwellings of the working classes, and in 1898 was appointed a C.B. in recognition of his long services in his important public posts.

Of forensic medicine and toxicology Cameron was long the acknowledged chief exponent and practitioner in Ireland, and his versatility was such that he for many years occupied the leading position in the country as both teacher of and practitioner in agricultural chemistry. His re-written edition of Johnston's earlier well-known work on this subject long occupied the position of a leading textbook. Among his many other contributions to scientific literature was a history of the Irish Royal College of Surgeons, his intimate association with which has been already indicated.

Few men can have had a larger circle of friends than Cameron or were more popular in Dublin society. He was delightful as a host and as a guest, and a charming talker with an ever-flowing fund of wit and humour, never exercised, however, at the expense of pain to others, for his kindness was even greater than his wit.

Apart from his numerous professional occupations, one of the main delights of his less strenuous hours was in Freemasonry, of which he was one of the most ardent and most honoured supporters in his native country.

But not the least of his achievements was the retention of his full mental faculties and his physical working powers beyond his ninetieth year.

BERNARD DYER.

ALEXANDER WYNTER BLYTH.

We deeply regret to record the loss of one of the original members of our Society, Dr. Alexander Wynter Blyth, whose death took place suddenly on March 31.

Wynter Blyth was the son of a medical practitioner, and was born in 1846 at Woolwich. He was educated for the medical profession at King's College, London, and became qualified in 1870. At a later period he studied law, and was called to the Bar as a member of Lincoln's Inn.

In 1878 he was appointed Public Analyst for Totnes, and subsequently for the County of Devon, in 1879 for Tiverton, and in 1882 Medical Officer of Health and Public Analyst for St. Marylebone, posts which he held until his death.

In the early days of the Food and Drugs Acts the appointments of Medical Officer and Public Analyst were frequently held by the same individual, and it was quite a common practice for medical men to spend a few months in the laboratory of a chemist to acquire some insight into the duties of what was then regarded as a subsidiary appointment.

Under such conditions it is not surprising that many of the early medical Public Analysts were not competent chemists, or that few improvements in the methods of chemical analysis originated with them.

Wynter Blyth was a striking exception, for having been trained as a chemist, he took an active interest in the affairs of the Society of Public Analysts from its inception.

A paper of his upon milk was published in 1876 in the *Proceedings*, which was the forerunner of the ANALYST. Numerous other contributions upon the analysis of foods and drugs will also be found in subsequent volumes of the journal. He served on the Council of the Society in 1877 and on three other occasions, and was thrice elected Vice-President, the last occasion being in 1895.

He also contributed to the *Transactions* of the Chemical Society, but he will be best remembered by his books on foods and poisons. Originally these formed parts of one volume, but in later editions the subjects were divided and published as separate works. The section on Foods was a comprehensive survey of the existing microscopical and chemical knowledge of the subject, and was one of the earliest attempts to systematise the modern methods of analysis. Its sixth edition in separate form was published in 1909 and reviewed in the ANALYST of that year. The second portion of the book included much information which was not readily accessible elsewhere, and as a manual of historical toxicology filled a distinct gap. Its fourth edition as a separate book was reviewed in the ANALYST for 1907, and a new edition has just been published.

As an instance of the unexpected detail to be found in this work, it may be recalled that in one of the actions arising out of the epidemic of arsenic poisoning of twenty years ago, it was pointed out that the possibility of glucose being a source of arsenical poisoning had there been mentioned.

It is interesting to note in this connection that Wynter Blyth was one of the earliest chemical investigators of cobra poison, and in 1878 received a Government grant in aid of his research.

He was elected a Fellow of the Institute of Chemistry in 1887 and served on its Council on two occasions. He was also Past President of the Incorporated Society of Medical Officers and Registrar of the Sanitary Institute. EDITOR.

SOREN HOY BLICHFELDT.

MR. SOREN HOY BLICHFELDT died on March 3, after a very short illness, at the age of forty-four.

Blichfeldt was born in Denmark, and received his chemical training at the Royal Pharmaceutical College in Copenhagen. His inclination towards fermentation problems then led him to the world-famous fermentation laboratories in Copenhagen, where he became one of Alfred Jørgensen's most valued assistants. Here he worked for several years on questions connected with the application of pure culture methods in the brewing and dairy industries, and while here he wrote a well-known Danish textbook on bacteriology.

In 1906 he joined the staff of the Maypole Margarine Works (then Messrs. Otto Monsted, Ltd.), and, as its chief technologist, was responsible for much of the development of the improved methods of margarine manufacture now in use. He remained with this firm until his death, at which time he was Assistant Managing Director.

He was always more interested in the application of science to industry than in pure science; hence his contributions to scientific literature lie mainly in the records of the Patent Office.

He was the originator of a distillation method for the estimation of mixtures of butterfat, coconut oil, and palm kernel oil, and a few days before his death his method for the determination of melting-points of fats was read before this Society.

He possessed a remarkable insight into the scientific problems underlying the industry with which he was connected, and his keenness and inexhaustible energy led to their rapid solution. His kindly tolerance and his generous spirit inspired a rare loyalty and enthusiasm in his staff, and his joyous disposition and entire freedom from affectation makes his loss all the greater.

T. THORNLEY.



LIQUID EXTRACT OF RED SQUILL (*SCILLA MARITIMA*) AS A RAT POISON.

By F. W. SMITH, B.Sc., A.I.C.

(*Read at the Meeting, February 2, 1921.*)

MR. BOULENGER, of the Royal Zoological Society, stated in an official pamphlet published in 1919, that an extract prepared from the bulb of the squill formed a most effective rat poison. It had the great advantage of being practically harmless to domestic animals. Opinions at the time appeared to be much divided as to the efficiency of the squill extract as a rat poison. It was decided, therefore, that

experiments should be undertaken in order to determine the toxic effect upon rats of various preparations of squill.

Samples of the various squill rat poisons on the market were found to differ to a very great degree. Their extractives varied from 2 per cent. to 15 per cent.

The results obtained with these preparations were most unconvincing, for out of five extracts considered, only one caused the death of a rat.

An aqueous extract (100 grms. drug—200 c.c. water) was prepared from the dried official squill (*Scilla urguinea*). The toxic effect of this extract upon rats appeared to be nil. It was noticed that this extract was not very bitter.

Kopaczewski (*Comptes rend.*, 1914) discovered that the toxic principle of squill was an amorphous glucoside, which he called "scillitin," and to which he assigned the formula $C_{17}H_{25}O_8$. This glucoside is sparingly soluble in water, but is quite soluble in alcohol. It is easily hydrolysed, yielding dextrose and amorphous products. It is also intensely bitter. The aqueous extract prepared was not bitter. In view of Kopaczewski's observation it would appear that scillitin was absent from the extract, particularly as scillitin is almost insoluble in water.

An aqueous extract prepared from the fresh white squill gave negative results.

From the results obtained with aqueous, alcoholic, and acetic acid extracts, it would appear that the glucoside, scillitin, which Kopaczewski said was most lethal to rats, was present in greater quantities in the red squill. The extract prepared from the dry red squill by means of alcohol also proved very effective; it was used three times, and proved fatal to rats in each case.

Other results suggest that the convenient method of preserving extracts by means of salicylic acid would not destroy the toxic principle of the squill, and could be adopted, without fear of hydrolysing the glucoside.

In the preparation of the extract, alcohol should be employed, otherwise the toxic glucoside would not be readily extracted. In the final form the glucoside appears to exist in solution in the aqueous liquid, as the alcohol is driven off.

During the first campaign against rats, the red squill was most difficult to obtain. Samples of alleged dried and fresh red squill were collected, and on three occasions proved to be white squill. They were easily identified by the absence of the deep red colour. It was observed that a section of red squill showed, under the microscope, the presence of isolated cells, at irregular intervals, filled with red colouring matter. These cells were not discovered in the white squill. A section of dried white squill coloured with vegetable dye did not show these isolated cells, but a continuous strip of colour. It is apparently impossible to disguise the white squill.

The non-toxic effect of some extracts and their unusual colour suggested the presence of artificial colouring matter in the white squill extract. Experiments were carried out to determine whether the addition of artificial colouring matter could be detected. It was observed that the extract of red squill gave, on evaporation to dryness, a residue which was of a homogeneous brown or brownish-red colour.

Extract of white squill, coloured with vegetable dyes, invariably yielded, on evaporation to dryness, a residue which was not homogeneous in colour, but showed bands of yellow round its outer edges. On spraying the residue with distilled water these yellow bands were more pronounced, owing to the greater solubility of the dye.

When coloured with aniline dye, this effect was not so marked, but on washing the residue, the distinctive yellow colour made its appearance.

In conclusion, I wish to thank Mr. F. J. C. Bird, of Messrs. C. R. Harker, Stagg and Morgan, Limited, in whose laboratory the initial experiments were conducted, and also Mr. T. J. Drakeley, M.Sc., for his kind encouragement and advice.

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DISCUSSION.

Mr. E. C. READ (*Technical Adviser, Ministry of Agriculture and Fisheries*) remarked that it might be assumed that scillitin was not, as Kobert had stated in his *Lehrbuch der Intoxicationen*, the only active principle in red squill. Preparations from the raw bulb soon lost their toxicity, especially in warm weather. The Pasteur Institute was originally responsible for the introduction of commercial preparations of red squill into this country, and at first they were intended to be used as supplementary in cases where rats and mice proved immune to the bacteriological "viruses."

Mr. C. L. CLAREMONT (*Ministry of Agriculture and Fisheries*) considered that Mr. Smith's experiments with aqueous extracts were unconvincing, since such preparations required diluting with milk and gruel to render them attractive. The whole of the toxic principle was not removed by extraction with alcohol, the residual powder left after extraction with 90 per cent. alcohol being more toxic than the alcoholic extract. It was possible that certain red squills on the market contained bulbs of *S. urguea Burkii*, which appeared to be less toxic than *Scilla maritima*. Chemical and microscopical examination were of little value in distinguishing between the three varieties of squills, and the actual administration to rats was the only ready means of identifying red squill in commercial preparations.

Mr. SMITH, in his reply, remarked that in view of his own experimental results, he could not understand the difficulty experienced in extracting the toxic principle with alcohol. Apparently the alcohol caused exosmosis to take place from the cells, and the glucoside was thus readily extracted.

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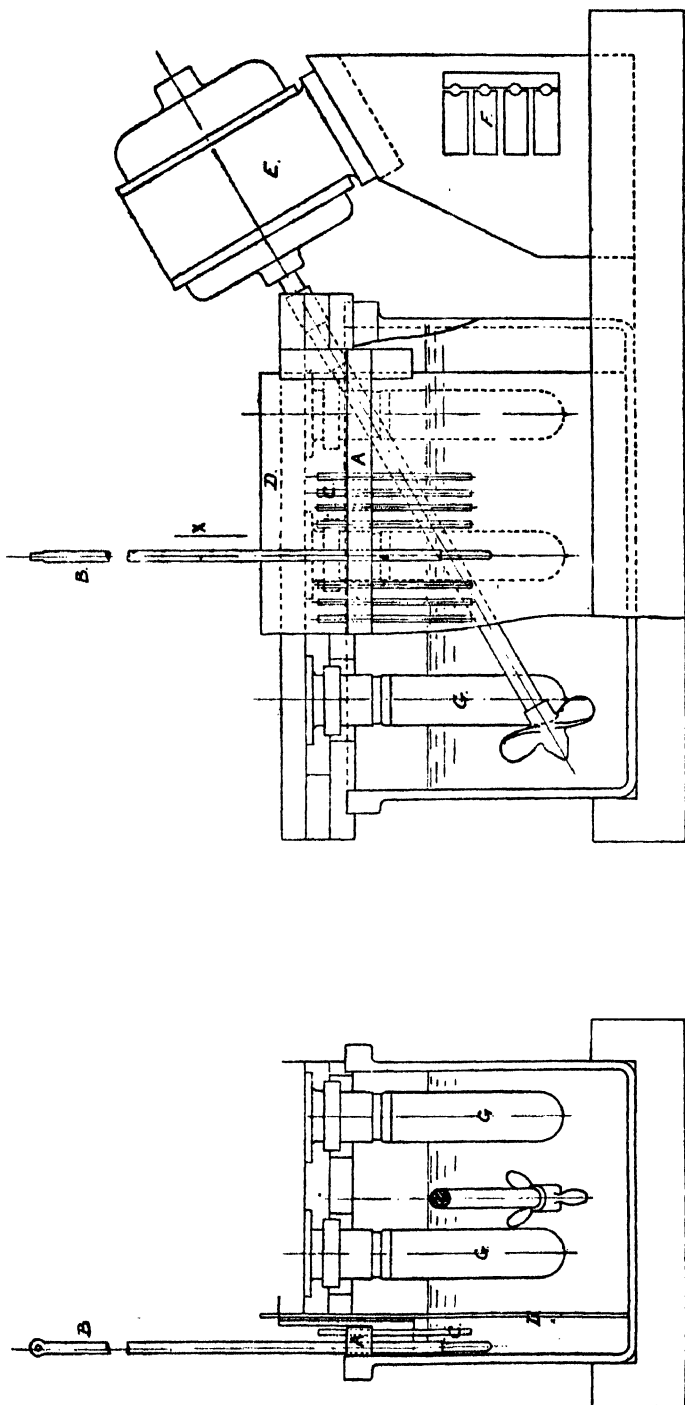
APPARATUS FOR THE ROUTINE DETERMINATION OF MELTING-POINTS OF FATS AND FATTY ACIDS.

By S. H. BLICHFELDT AND T. THORNLEY.

(Read at the Meeting, March 2, 1921.)

A NEED has long been felt for a method of determining melting-points of fats which shall be rapid and accurate, giving strictly comparable and reproducible results, and capable of application by a relatively unskilled operator to a number of samples at one time.

It is difficult accurately to define the melting-point of such a complex mixture of glycerides as a fat. The melting-point varies enormously with the method of



SECTION ON XY

A SUPPORT FOR THERMOMETER AND TUBES.

B. THERMOMETER.

C TUBES.

G HEATING LAMP.

D. OPAQUE GLASS SHEET.

E. MOTOR DRIVING PROPELLER.

F. PLUGS FOR CONNECTING UP LAMPS + MOTOR TO MAINS.

preparation of the sample and with the method of carrying out the actual determination, and there is no doubt that many of the wide variations in the recorded melting-points are due to divergences in methods and determination.

By "melting-point" in this method is understood the temperature at which a column of fat of specified dimensions begins to move in an open tube of specified dimensions under a definite hydrostatic pressure.

METHOD.—A clean glass tube 6.5 cm. long, 1 mm. bore and 3 mm. diameter, is dipped into the melted mixed sample. The fat is allowed to rise just over 1 cm. in the tube, and the lower end of the tube is then brought into contact with filter paper or any absorbent material, and just so much melted fat is withdrawn that the column remaining is 1 cm. long. The column of completely liquid fatty material is solidified by placing the tube between two blocks of ice, where it remains during two hours.

The tube is then fixed vertically in a water bath capable of being slowly and regularly heated with constant stirring. The tube is immersed so far that the upper surface of the fat is 1 cm. below the level of the water. The water is heated so that the temperature rises 1° C. per minute, and the temperature at which the fat begins to slide up the tube is noted as the melting-point.

The apparatus shown is adapted for the simultaneous determination of melting-point of a number of samples. A glass tank carries a hard cover, on the under side of which are mounted sockets for six 16 c.p. carbon filament lamps. These are connected in three pairs with plug sockets, so that two, four, or six lamps may be connected with the supply. The fourth plug switches in the motor driving the stirring propeller.

The cover supports vertically a sheet of black or dark-blue glass, which divides the tank into two compartments. The large compartment contains the heating bulbs and propeller, and the smaller one the sample tubes and thermometer. The sheet does not quite reach the ends of the tank, so that, when the propeller is running, a rapid current of water flows past the tubes without too much commotion on the surface. Sufficient light is reflected round the ends of the sheet to provide ample illumination of the tubes. The bar carrying the tubes is supported by a pair of ebonite brackets, and the thermometer is slung by wire hooked over the top of the glass sheet.

By adherence to such a method, the wide divergences in melting-points of fats indicated by the published figures seem to be considerably reduced.

The melting-points of butter fat, oleo, and palm oil samples, determined after rapid and slow cooling are given in the following table :

				Quick Cooling. Two Hours in Ice.	Slow Cooling. Twenty-four Hours at Room Temperature.
				° C.	° C.
Butter	...	...	...	31.0	32.2
Oleo	...	...	...	29.3	32.1
Palm oil	...	...	...	35.0	37.4

THE DETECTION OF ADULTERATION IN BUTTER BY MEANS OF THE MELTING-POINT OF THE INSOLUBLE VOLATILE ACIDS.

By GEORGE VAN B. GILMOUR, B.Sc. (Lond.), A.R.C.Sc.I., A.I.C.

(Read at the Meeting, March 2, 1921.)

USE has already been made of the melting-points of the insoluble volatile acids from coconut and palm kernel fats for distinguishing between these fats in margarines and other mixtures (see Blichfeldt, *J. Soc. Chem. Ind.*, 1919, **38**, 150-152r). The object of this investigation was to determine the melting-point of the insoluble volatile acids from butter fat, to see how this varies with different varieties, and to find out if the determination of the melting-point would be useful in detecting the presence of coconut and palm kernel fats in butter.

The term "melting-point," wherever used in this paper, refers to the melting-point of the insoluble volatile acids.

The melting-point determinations were made upon the insoluble volatile acids obtained by using the Blichfeldt distillation apparatus, the procedure being as follows: After the distillation is completed, the apparatus is disconnected and the acids transferred while still warm to a clean test-tube. This is done by manipulating the apparatus so that the insoluble acids are first introduced into the upper bulb and then run out into the test-tube through the side delivery tube. The test-tube is filled almost to the top with warm liquid from the apparatus, and then gently rotated in the hand so that all the globules of fatty acids rise to the surface and coalesce to form a large globule. If the test-tube is not clean the globules have a tendency to stick to the walls of the tube.

The capillary tube method for determining melting-points as described by Blichfeldt and Thornley (*ANALYST*, 1921, p. 180) was that used. The acids being at the top of the test-tube, a column can easily be introduced into the capillary tube by immersing one end of it in the acids.

Occasionally the quantity of insoluble volatile acids is so small that difficulty is experienced in getting a column to rise in the capillary tube without some water rising at the same time. This can be overcome by solidifying the acids and removing them from the test-tube to a watch-glass. After drying by touching them with a piece of filter paper, the acids are melted and a column introduced into the capillary tube.

In the first place it must be pointed out that it is just as important in determining the melting-point that the distillation should be carried out in exactly the same way as when estimating the soluble and insoluble volatile acids, because the melting-point depends on the quantity of acids that come over. This will be apparent when the following figures are considered where the melting-points of the insoluble volatile acids obtained from three successive distillations of the fatty acids from 5 grms. of fat are shown both in the case of coconut and palm kernel fats. The second and third distillations were carried out by adding 100 c.c. of water

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to the distilling flask after the first or second distillations were finished, and distilling to the same mark as before.

	Coconut Fat.	Palm Kernel Fat.
	° C.	° C.
First distillation	10·2	23·4
Second „	19·6	28·3
Third „	26·0	32·0

The melting-points for a number of butters obtained from different sources are given below; in this table are shown the distillation figures of these butters determined by the author's method (ANALYST, 1920, 45, 2-7), also the corresponding figures for the coconut and palm kernel fats used in the mixtures referred to later.

Sample.	Total Volatile Acids.	Soluble Volatile Acids.	Insoluble Volatile Acids.	Melting-Point of Insoluble Volatile Acids.
				° C.
Butter 1	31·0	22·6	8·4	15·8
2	30·4	22·3	8·1	16·0
3	30·3	22·9	7·4	16·8
4	29·2	22·1	7·1	17·9
5	28·7	22·2	6·5	19·5
6	29·8	23·4	6·4	20·0
7	28·8	22·5	6·3	20·3
8	26·3	20·9	5·4	25·6
9	26·5	21·2	5·3	21·7
10	25·4	20·3	5·1	22·1
11	27·7	21·8	5·9	23·0
12	29·4	22·4	7·0	22·3
Coconut fat	21·1	1·6	19·5	9·9
Palm kernel fat	13·7	1·1	12·6	21·2

The limits between which the melting-point can lie are fairly wide apart. In the above table the limits are 15·8° and 25·6° C. Early results seemed to indicate that the melting-point was inversely proportional to the quantity of the insoluble volatile acids present, but later determinations did not bear this out. It can, however, be said of the butters examined that those with high melting-points usually had low distillation figures, and *vice versa*.

From the behaviour of the melting-points of the insoluble volatile acids derived from mixtures of coconut and palm kernel fats (Blichfeldt, *loc. cit.*), where these melting-points are stated to lie between the melting-points of the acids from the pure fats and are approximately proportional to the amounts present, it was thought that similar results would be obtained by the addition of either of these fats to butter,

That this is not the case can be seen from the figures below, showing the melting-points of the acids from butters which contain coconut and palm kernel fats.

Butter Sample.	Butter + 10 per Cent. Coconut Fat.		Butter + 10 per Cent. Palm Kernel Fat.	
	Melting-point of Insoluble Volatile Acids.	Total Volatile Acids. (Calculated.)	Melting-point of Insoluble Volatile Acids.	Total Volatile Acids. (Calculated.)
	° C.		° C.	
1	10.5	30.0	13.0	29.3
2	12.1	29.5	13.7	28.7
3	11.5	29.4	13.1	28.6
4	12.5	28.4	16.1	27.6
5	10.9	27.9	14.2	27.2
6	12.0	28.9	15.1	28.2
7	12.1	28.0	16.8	27.3
8	15.2	25.8	18.1	25.0

Butter Sample 2, containing 5 per cent. coconut fat + 5 per cent. palm kernel fat, gave a melting-point of 11.3° C. Total volatile acids calculated = 29.1.

Butter Sample 4, containing 5 per cent. coconut fat + 5 per cent. palm kernel fat, gave a melting-point of 14.1° C. Total volatile acids calculated = 28.0.

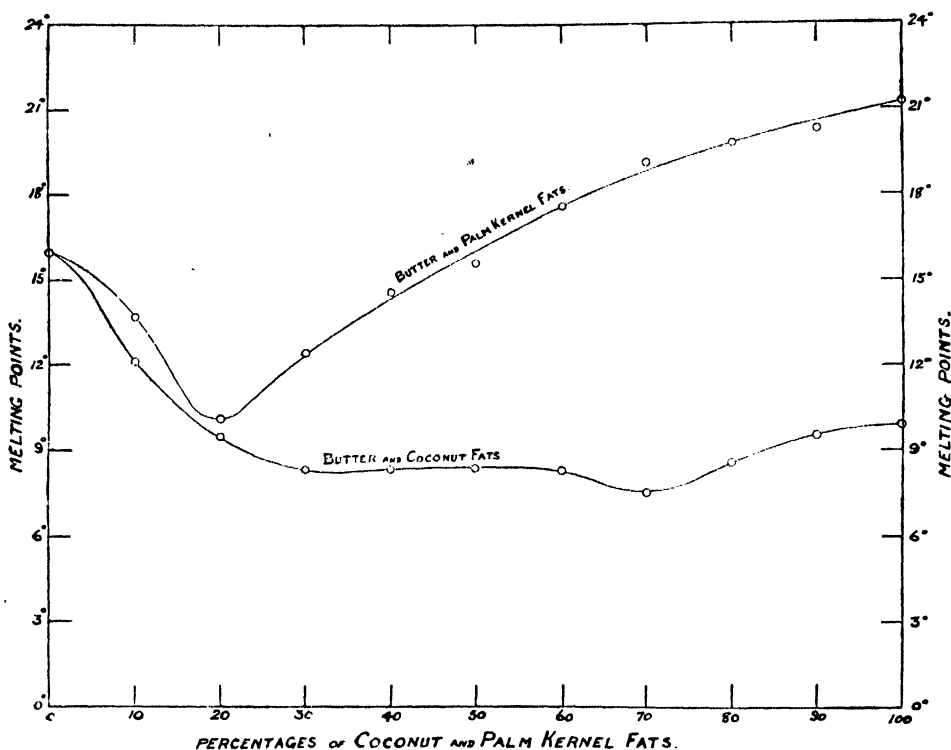
Butter Sample 7, to which was added 10 per cent. of margarine containing 80 per cent. of a mixture of coconut and palm kernel fats, gave a melting-point of 15.4° C. Total volatile acids calculated = 27.3.

The butters numbered 1 to 8 in the mixtures are the same as those bearing similar numbers in the previous table.

Per Cent. Butter (2).	Per Cent. Coconut Fat.	Melting-Point of Insoluble Volatile Acids.	Per Cent. Butter (2).	Per Cent. Palm Kernel Fat.	Melting-Point of Insoluble Volatile Acids.
		° C.			° C.
100.0	0.0	16.0	100.0	0.0	16.0
90.0	10.0	12.1	90.0	10.0	13.7
80.0	20.0	9.5	80.0	20.0	10.1
70.0	30.0	8.3	70.0	30.0	12.4
60.0	40.0	8.3	60.0	40.0	14.6
50.0	50.0	8.3	50.0	50.0	15.6
40.0	60.0	8.2	40.0	60.0	17.6
30.0	70.0	7.5	30.0	70.0	19.2
20.0	80.0	8.5	20.0	80.0	19.8
10.0	90.0	9.5	10.0	90.0	20.3
0.0	100.0	9.9	0.0	100.0	21.2

If the table of melting-points of the acids from pure butters be examined it will be seen that all the butters with figures for total volatile acids below 28 have

melting-points above 20.0°C ., and no exception has so far been met with. Apparently, then, any butter with a total volatile figure below 28 and a melting-point below 20.0°C . can be assumed to be adulterated. In most of the above examples, where coconut or palm kernel fats, or both, were added to butter fat, the melting-points of the acids from the mixtures fell below 15.8°C ., which is the minimum for pure butter. In the case of those examples where the melting-points were above 15.8°C ., the total volatile figures were below 28, and, as none of the melting-points reached 20.0°C ., it follows that adulteration has been detected in every case.



The effect on the melting-point when increasing quantities of coconut and palm kernel fats are added to butter fat was studied with the results given on p. 185.

A graphical representation of these results is shown in the diagram.

The addition of increasing amounts of coconut fat to the butter fat caused the melting-point to fall rapidly at first until there was about 25 per cent. present; then it remained fairly steady up to 60 per cent. At this point it began to fall again, reaching a minimum of 7.5°C . when there was 70 per cent. of coconut fat present. Further additions now raised the melting-point steadily until that of pure coconut fat was reached.

In the case of palm kernel fat the melting-point fell rapidly until about 20 per cent. was present, when the lowest melting-point—viz., 10.1°C .—was obtained. On

further addition, the melting-point steadily rose until that of pure palm kernel fat was attained.

The corresponding graph for mixtures of the insoluble volatile acids from coconut and palm kernel fats should be a straight line joining the melting-points of the acids from the pure fats. It can thus be assumed that the insoluble volatile acids from coconut and palm kernel fats are very similar in composition, but that those from butter fat differ considerably. This difference consists probably in the relative proportions of caprylic, capric, and lauric acids present.

The eutectic point on the graph for mixtures of palm kernel and butter fats is much more decided than that for coconut and butter fats.

The graphs also showed that the melting-points of the acids from a margarine containing a high percentage of coconut and palm kernel fats will be only slightly affected by the addition of 10 per cent. of butter, but that if the margarine contain a small percentage of these fats the addition of 10 per cent. of butter will have a more marked effect.

CONCLUSIONS.—The determination of the melting-point of the insoluble volatile acids is useful as a confirmatory test in the analysis of butter fat.

When the melting-point is taken into consideration with the figure representing the total volatile acids, it should give sufficient evidence of the purity of the sample. If the figures point to the sample being pure, the volatile acids need not be separated into soluble and insoluble fractions.

The melting-points for pure butters lie between 15.8° and 25.6° C. Butters with high total and insoluble volatile figures usually have low melting-points, and *vice versa*. Genuine butters give melting-points above 15.8° C., but if the total volatile figure falls below 28, then the melting-point should be above 20.0° C.

In many cases the melting-point will give an indication of adulteration when distillation methods fail to do so. In the case of a butter, for instance, with high distillation figures and a low melting-point, if adulterated with a fat which does not give rise to volatile acids the volatile acids will be reduced in proportion to the amount of adulteration, but not necessarily below the legal limit, whilst the melting-point will continue to represent the figures that the pure butter would give, and will probably be too low for the reduced total volatile figure.

The addition of either coconut or palm kernel fats to butter fat has the effect of lowering the melting-point of the insoluble volatile acids.

The melting-point will readily detect 10 per cent. of coconut or palm kernel fat. Smaller quantities can also be detected, but not with the same certainty.

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THE ESTIMATION OF STRYCHNINE IN SCALE PREPARATIONS CONTAINING QUININE AND OTHER CINCHONA ALKALOIDS.

By T. F. HARVEY, F.I.C., AND S. BACK.

(*Read at the Meeting, April 6, 1921.*)

THE estimation of strychnine in a scale preparation containing iron and quinine is a matter of some difficulty. The obvious method would be to extract the total alkaloids and then separate them by a reliable method.

The chief methods that have been proposed for the separation of these two alkaloids are :

1. Precipitation of the quinine as oxalate. The separation is not complete and the method does not appear to have been much used. Applied to the total alkaloids obtained from Easton's syrup this method gave a crude residue containing three and a half times as much quinine as strychnine.

2. A method published in the *J. Amer. Pharm. Assoc.*, 1919, 8, 804-807, which is said to depend on the solubility of strychnine in water and its slight solubility in ether.

This method is not only cumbersome, involving the manipulation of considerable volumes of liquid and fourteen separations, but appears to be theoretically unsound, since strychnine is more soluble in ether than in water, and will partition itself between the two solvents according to well-established laws. However, we carried out an experiment using 475 mgrms. quinine and 25 mgrms. strychnine and extracted with nine consecutive quantities of ether of 8 c.c. each from a volume of 222 c.c. aqueous phase. The quinine fraction weighed 461 mgrms., from which we recovered 7 mgrms. of strychnine, whilst the strychnine fraction weighed 37.7 mgrms. but contained only 15.3 mgrms. strychnine.

We have found the solubility of powdered strychnine in ether of 0.720 sp. gr. at 15° C. to be 0.034 gm. per 100 grms. of solution, whilst Seidell's Solubilities gives 0.0432 gm. at 20° C., and 0.0513 gm. for wet ether. Taking our own figure as a basis, and the solubility of strychnine in water as 1 in 6,420, it can be calculated that the amount of strychnine remaining in the aqueous phase after the above treatment with ether should be 15.27 mgrms., which by coincidence is in exact agreement with the amount found.

3. Precipitation of quinine as tartrate with subsequent recovery of the strychnine and final washing with ether. (Harrison and Gair, *Yearbook Pharm.*, 1903, 564.) Harrison and Gair obtained very good results with mixtures of pure alkaloids, which were not seriously affected by the presence of other cinchona alkaloids occurring in the quinine sulphate of commerce. We find that this method gives quite good results on known mixtures of the pure (B.P.) alkaloids, as the figures in the table show. When, however, the method was applied to the dried alkaloidal residues obtained from iron, quinine, and strychnine citrate, results much in excess of the truth were obtained, and the method was obviously unsuitable for the purpose. The unwashed

strychnine (?) residues in these cases amounted sometimes to about 180 mgrms. instead of 50 mgrms., and it is impossible to complete the separation of the two alkaloids in a residue of this nature by means of ether. Washing with ether should only be resorted to for removing a comparatively small amount of impurity from a strychnine residue.

	Mgrms.	Mgrms.	Mgrms.
Quinine alkaloid taken	750	750	750
Strychnine taken	50	50	50
Unwashed strychnine obtained	59	60	56
Strychnine, after washing with 3 × 1 c.c. washed ether	53	51	51

4. A fourth method of separating these two alkaloids is that of Simmonds (ANALYST, 1914, 39, 81). It depends on precipitation of strychnine ferrocyanide from 10 per cent. sulphuric acid solution followed by reprecipitation of the strychnine salt from 20 per cent. sulphuric acid.

We obtained the following results by Simmonds' method, working on pure alkaloids and using always 750 mgrms. dry quinine and 50 mgrms. strychnine :

Strychnine taken	50 mgrms.	50 mgrms.
Time of standing with ferrocyanide	21 and 23 hours	2½ and 2 hours
Strychnine residues obtained ...	56 mgrms.	54 mgrms.

These results, which are 12 and 8 per cent. too high, are probably due to the initial presence of a rather high concentration of quinine (750 mgrms. in 50 c.c.), but whatever the cause, the errors are large and even by washing carefully three times with 1 c.c. of washed ether as in Harrison and Gair's method they are only reduced to 6 per cent. Other experiments introducing slight variations have given similar results. When Simmonds' method is applied to the alkaloidal residue obtained from iron, quinine, and strychnine citrate, it is more unsatisfactory, and we found it necessary to seek some modification or combination of methods on the results of which one could rely with some confidence. It is obvious that the extracted and dried residue from scale preparations will differ in some degree from the pure alkaloids themselves, quinine being very prone to slight alteration during the processes of manufacture and analysis. It is therefore preferable to separate the strychnine from an undried alkaloid, and to make a separate estimation of the total alkaloid.

The method which has given the best results is a combination of the methods of Harrison and Gair and of Simmonds, but we found that the tartrate could be conveniently precipitated from the solution of the scales in water without previous isolation of the alkaloids. This direct precipitation of quinine has proved easy, and incidentally avoids shaking out with solvents at a stage at which emulsification is very prone to occur. To save time and avoid washing the bulky quinine tartrate, we adopted the practice of adjusting the total volume and taking an aliquot part of the filtrate, making the necessary percentage correction at the end.

The method is as follows, the time required for the actual manipulation being about two and a half hours. In the case of an iron quinine and strychnine scale preparation containing about 15 per cent. of quinine and 1 per cent. of strychnine, 10 grms. are dissolved in 70 c.c. of water in a beaker, 5 c.c. of $\frac{N}{1}$ sulphuric acid added, and the whole almost neutralised with ammonia, until the precipitated quinine only just redissolves. Thirty grms. of Rochelle salt are added, and the liquid is neutralised with dilute ammonia, the final reaction being left faintly acid to litmus paper. The mixture is stirred and heated in a water bath for fifteen minutes, cooled and transferred to a 100 c.c. measuring flask, the beaker being rinsed with water so as to make the volume up to 100 c.c. After standing for two hours the liquid is filtered, the first 10 c.c. being rejected. Fifty c.c. of the filtrate are extracted three times with chloroform, using 30 c.c., 10 c.c., and 10 c.c., and ammonia, and the mixed chloroform solutions are washed twice with 5 c.c. of water. The chloroform is extracted with 30 c.c. of 10 per cent. (weight/vol.) sulphuric acid, and twice more with 10 c.c. of the same acid, the acid liquids being collected in a small (60 c.c.) separator previously plugged with a small piece of cotton-wool. Five c.c. of freshly made 4 per cent. potassium ferrocyanide solution are added, the separator is practically filled with 10 per cent. acid (to exclude air), and after rotation the whole is allowed to stand in a dark place for two hours. It is advisable to be sure that precipitation has definitely occurred before placing aside. We prefer to use 10 per cent. acid in the precipitation. Simmonds makes use of 20 per cent. acid, but in that case the precipitation is very sluggish, and two hours' standing might be insufficient. At the end of this time the acid is forced out through the plug, the strychnine ferrocyanide washed twice with 3 c.c. of 5 per cent. sulphuric acid, and the strychnine recovered by shaking with 10 c.c. of 10 per cent. ammonia, and 15 c.c., 10 c.c., and 10 c.c. of chloroform.

Evaporation is carried out as usual, an addition of about three drops of amyl alcohol being made towards the end to prevent decrepitation of the strychnine crystals. When cold, the alkaloidal residue is washed three times with 1 c.c. of ether and dried at 100° C. It usually consists chiefly of characteristic crystals of strychnine, which are of a pale brown tint.

A correction of -1.7 per cent. has been established for the volume of the quinine tartrate when following the above procedure. The following experiments seem to show the accuracy of the method for iron quinine and strychnine citrate.

A sample of iron and quinine citrate precipitated as described with tartrate left 160 mgrms. of alkaloid unprecipitated. This, on precipitation with ferrocyanide, yielded 2 mgrms. of residue which was entirely dissolved by the ether used for washing. One hundred mgrms. of strychnine were dissolved in $\frac{N}{1}$ acid and added to a solution of 10 grms. of the above iron and quinine citrate. The strychnine was then estimated as above. The weight of unwashed strychnine obtained was 50.5 mgrms., which, after washing with ether, weighed 49.0 mgrms. or 48.2 mgrms. (corrected). (Theory = 50 mgrms.)

Another experiment with a different sample of iron and quinine citrate gave 49.7 mgrms. (corrected).

Several commercial samples of iron quinine and strychnine citrate gave the

following results as compared with the expected 1 per cent. : 0.94, 0.94, 0.92, 0.94, 1.00 and 1.04 per cent.

The occurrence of other cinchona alkaloids as impurities has received some consideration, the results of which are shown in the table below.

Quinidine is the least likely to occur, but if present would, together with cinchonine, escape precipitation as tartrate. Quantities of 150 mgrms. of each of these alkaloids were dissolved separately in 10 per cent. and in 20 per cent. sulphuric acid and treated with potassium ferrocyanide solution. In no case did any precipitation occur during two hours' standing, and it is therefore not likely that small quantities of these alkaloids would introduce any inaccuracy. Cinchonidine, the most usual impurity in quinine salts, on the other hand, is very completely precipitated as tartrate. For instance, 300 mgrms. of cinchonidine were precipitated as tartrate under the above experimental conditions, and the amount of unprecipitated cinchonidine was only 4 mgrms., which, when subjected to the ferrocyanide precipitation, gave no trace of precipitate within two hours. The table shows that 150 mgrms. of cinchonidine dissolved and treated directly with ferrocyanide solution cause a considerable amount of crystalline precipitate to appear. On the other hand, we have found that 50 mgrms. of strychnine with 75 mgrms. of cinchonidine are easily and completely separated by Simmonds' method (two precipitations with ferrocyanide in 10 and 20 per cent. sulphuric acid). It is therefore highly improbable that any trace of cinchonidine sufficient to interfere with the method could get through from the tartrate precipitation.

There is left the possibility of the occurrence of quinicine as an alteration product of quinine. This alkaloid is hardly likely to occur in any quantity. It is, however, not precipitated by neutral tartrate, and is thrown down by ferrocyanide together with the strychnine. Its presence is made fairly obvious by the yellow colour of its acid solutions, and by the dark and abnormal appearance of the strychnine residue.

We have treated by the above method two alkaloid mixtures, with the results shown below :

Alkaloids taken.		Unwashed Strychnine Residue.	Ether-washed Residue.
		Mgrms.	Mgrms.
Strychnine, 50 mgrms.	} ...	73	71.5
Quinicine, 37½ "			
Strychnine, 50 "	} ...	63	61
Quinicine, 37½ "			
Quinine, 712½ "			

Thus, although dry quinicine is very soluble in ether, it is not removed from such residues by this means, and we have found by experiment that pure quinicine dissolves only slowly in ether. Attempts to soften the residues with alcohol and then treat with ether were not successful, far too much strychnine being removed. Experiments were then made with pure acetone, in which strychnine dissolves to the extent of 0.132 gm. per 100 c.c. (1 in 750), whilst quinicine dissolves both rapidly and completely in all proportions.

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It will be seen from the accompanying figures that 10 mgrms. of quinicine can be separated from 50 mgrms. of strychnine by carefully washing the mixture twice with 0.5 c.c. of acetone, whilst in the presence of 20 mgrms. of quinicine the results obtained by washing are of very doubtful value.

Quantity Alkaloids taken.	Weight in Mgrms. after Washing with Pure Acetone.		
	2 × 0.5 c.c.	3 × 0.5 c.c.	5 × 0.5 c.c.
	Mgrms.	Mgrms.	Mgrms.
Strychnine, 50 mgrms. }	49.0	—	—
Quinicine, 10 " }			
Strychnine, 50 " }	58.5	{ 54.0	50.5
Quinicine, 20 " }		{ 54.5	

Solubility at 15° C. in grms. per 100 c.c. of Solution.

	Ether, Sp. Gr. 0.720.	Acetone.
Strychnine	0.244	0.132
Quinicine	Very soluble (slowly).	Soluble in all proportions.
Quinine	—	2.32

The authors are indebted to Messrs. Thomas Morson and Son, Ltd., in whose laboratory this investigation was made.

BEHAVIOUR OF CINCHONA ALKALOIDS WITH FERROCYANIDE IN ACID SOLUTION.

Alkaloid.	Mgrms. taken.	Strength Acid (Per Cent. by Volume).	Immediate Result.	Result after Two Hours in Dark.
Quinidine ...	150	{ 10	Nil.	Nil.
		{ 20	"	"
Cinchonine ...	150	{ 10	"	"
		{ 20	"	"
		{ 10	"	Considerable crys-
Cinchonidine ...	150	{ 20	"	talline ppt.
		{ 10	"	Smaller ppt. than in
Cinchonidine ...	100	10	"	10 per cent. acid.
			"	Few very small crys-
				tals (after 12 hours).
	{ 20	{ 10	Nil (ppt. in 5 mins.)	Heavy ppt.
		{ 20	Distinctly opalescent.	" "
Quinicine ...	{ 40	{ 10	Opalescence (ppt. in	" "
		{ 20	5 mins.).	
		{ 20	Distinct ppt.	" "
	{ 150	{ 10	Immediate ppt.	Considerable ppt.
		{ 20	" "	(About 50 per cent.
				pptd.).

(The substance was dissolved in 50 c.c. of the sulphuric acid and 5 c.c. of recently made 4 per cent. potassium ferrocyanide solution were added with rotation.)

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- Harrison and Gair, *Year-Book of Pharmacy*, 1903, 564. Simmonds, *ANALYST*, 1914, 81. Allen's "Commercial Organic Analysis," VI., 461, 462, 543; IX., 518.
Quinicine: Pasteur, *Jahresberichte*, 1853, 473. D. Howard, *J. Chem. Soc.*, 1871, 24, 61; 1872, 25, 101. Howard and Chick, *J. Soc. Chem. Ind.*, 1909, 54. Henry, "Plant Alkaloids," 147.



A COLOUR REACTION FOR ACONITE.

By S. MALLANNEH, M.D., D.P.H.

(*Read at the Meeting, April 6, 1921.*)

THE colour reactions at present known are not reactions of aconitine but of benzoic acid, which is one of the products of decomposition of aconitine. Hence the colour reactions are not specific.

Most of the colour reactions for alkaloids are such that they are useful only when applied to pure samples of alkaloids, but they are of no use when applied to crude substances containing alkaloids.

In medico-legal cases, especially in India, the poisoning is generally caused by the administration of crude substances in the form of powdered root, bark, or seeds, and not by the use of active principles. If the quantity of the vegetable poison present in the stomach be small, as is generally the case with aconite, it is next to impossible in most cases to get a sufficient amount of alkaloid extracted in order to prove the presence of poison by means of experiments on animals.

In India the vegetable poison also undergoes decomposition so quickly that it is almost impossible to detect its presence in a dead body, though distinct clinical symptoms of poisoning might have been present before death. But the cause of the failure to detect the poison in such cases is that, up to date, there is no reliable chemical test known for aconite.

As the result of my experiments, I have discovered a test which I think is very useful for medico-legal purposes. If a minute particle of potassium ferricyanide be placed close to a minute portion of aconitine or a small portion of powdered root of aconite, and then a drop of formic acid added, a green coloration immediately appears. This is a very delicate reaction, as $\frac{1}{1000}$ grain of aconitine gives a positive reaction. Heat should not be applied for this test.

Morphine, atropine, digitalin, strychnine, eserine, and hyoscyamine do not react to this test, which therefore seems to be specific for aconite. It is not only applicable to the pure alkaloid, but also applicable to powdered root of aconite. Hence, if confirmed, it will be of great toxicological importance. Recently, in a case of human poisoning at Banswada Nizamaabad, I was able to test this reaction. A few black fragments found adherent to the stomach wall of the deceased gave positive reaction

to this test and the case was confirmed subsequently by experiments on animals. The police were able to procure from the house of the culprit a brown powder, which on examination was found to be powdered root of aconite.

DISCUSSION.

Mr. H. FINNEMORE said that, apart from the limited value of all colour reactions, this test was not specific for aconitine, since it appeared to be given by the Indian variety of aconite, which contained pseudaconitine but not aconitine.



A METHOD FOR THE DETERMINATION OF THE ACIDITY OF COLOURED SOLUTIONS.

By J. L. LIZIUS, B.Sc., A.I.C.

(Read at the Meeting, April 6, 1921.)

THE filter paper of a small Buchner funnel (about 2 inches in diameter) is moistened with several drops of phenolphthalein solution. The pump is turned on, and when the excess of phenolphthalein has been removed the pump is turned off again. To 10 c.c. of the coloured solution $\frac{N}{10}$ alkali is added until it is just alkaline. The test for alkalinity is made by withdrawing a little of the solution by means of a fine capillary tube (a melting-point tube), and touching the filter-paper. The liquid remaining in the capillary tube is blown back into the solution. When the capillary tube test gives a pink colour the whole solution is run through the Buchner funnel. The filter-paper is left a pink colour. The contents of the Buchner flask are washed out into a beaker and 0.05 c.c. of $\frac{N}{10}$ acid is added. One or two drops of phenolphthalein solution are run on to the filter-paper, and the coloured solution is again poured through. If the filter-paper is not pink the volume of $\frac{N}{10}$ alkali added is the titration value. If the filter-paper is still pink the addition of $\frac{N}{10}$ acid is continued, 0.05 c.c. at a time, until the filter paper is no longer pink. The titration value is the volume of $\frac{N}{10}$ alkali—(volume $\frac{N}{10}$ acid - 0.05 c.c.). If the colour of the solution is adsorbed by the filter and tends to mask the pink colour, the difficulty can be overcome by washing through the paper a drop or two of phenolphthalein solution. Coloured alkaline solutions can be titrated by $\frac{N}{10}$ acid in a similar manner.

The pink colour left on the filter paper rapidly fades, owing to the action of the carbon dioxide in the air.

This method was tested by four different observers on a sample of stout. The results obtained by the above method agreed to within 0.1 c.c. $\frac{N}{10}$ alkali, whereas by the ordinary method of titration the results varied over 0.6 c.c.

In the case of blue-black inks, the determination is extremely difficult for the following reasons:

1. The ink itself acts as an indicator, becoming violet before the neutral point is

reached. The combination of the pink colour of phenolphthalein in alkaline solution with the colour of the ink also produces a violet colour.

2. The colour of the ink is very strongly adsorbed by the filter-paper, and this colour is not readily washed out by phenolphthalein. The method of decolourising with hydrogen peroxide (ANALYST, 1921, 131) is much simpler and more satisfactory for inks.

I have no doubt that the method will prove suitable for very dark vinegars and for molasses, as it has been shown suitable for stout. I have correctly determined the acidity of a sample of vinegar, and of a solution of hydrochloric acid to which caramel had been added, in this way.

The note is put forward as a more satisfactory method for the acidity of the coloured solutions the analytical chemist has to determine, but does not claim to be a general method of determining the acidity of every conceivable coloured solution.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Wet and Dry Feeding of Concentrates to Dairy Cows. R. A. Berry. (*J. Agric. Sci.*, 1921, 11, 78-98.)—The effect of wet and dry feeding has been investigated on a herd of ten Ayrshire cows with special reference to the effect on health, condition, milk yield, and fat content. Water consumption and milk yield and temperature effects have also been studied. Feeding with wet meal for a period of five weeks raised the milk yield by about $\frac{3}{4}$ lb. per cow per day, and the fall in yield due to advanced lactation was reduced. On a ten weeks' wet feeding the yield is increased, but the advantage diminishes as lactation advances. Cows fed on wet food take in altogether more water *per diem*, the exact ratio of water consumed varying with the fodder, and increasing as the milk yield increases, the latter representing about 26 per cent. of the total intake. On a heavy-root feeding the milk yield decreases, but the fat content rises materially, the non-fatty solids remaining constant. The health of the cows is adversely affected. The mean temperature in the byre is 15° to 20° F. above the outside temperature; lowering the temperature decreases the milk yield and *vice versa*, and it has been shown (*cf.* Berry, *Bull.* 76, *W. Scot. Agric. Coll.*, 1916, 49) that the butyric acid content is reduced by low temperatures.
H. E. C.

Coffee Substitutes from Narcissus Bulbs and Seaweed. C. Griebel and W. Rothe. (*Zeitsch. Unters. Nahr. Genussm.*, 1921, 41, 69-73.)—Roasted narcissus bulbs have been used in Germany as a coffee substitute; the raw bulbs contain

an alkaloid, which acts as an emetic, but in a case of sickness supposed to be caused by the consumption of this coffee substitute the authors were unable to detect any trace of the alkaloid, possibly because it had been decomposed during the roasting process. The ground, roasted bulb has a characteristic appearance under the microscope, the long clusters of calcium oxalate crystals being particularly conspicuous. Another coffee substitute is prepared from seaweed (*Zostera marina*); this may also be identified by microscopical examination. W. P. S.

Poisonous Haricot Beans. Th. v. Fellenberg. (*Chem. Zeit.*, 1921, **48**, 201.)—The striated markings, or the network pattern, in the European variety of the bean *Phaseolus lunatus* can be easily distinguished if the beans are treated with basic dyes, such as fuchsin and methyl violet. The poisonous properties of the beans may be destroyed by boiling them, but such a course does not always secure complete immunity. W. J. W.

Adulteration of Ground Cinnamon with Ochre. R. Windisch. (*Zeitsch. Unters. Nahr. Genussm.*, 1921, **41**, 78-81.)—Of nine samples of ground cinnamon examined, four yielded from 6 to 10 per cent. of ash, having a red-brown colour, and containing up to 4 per cent. of sand. Genuine cinnamon gave less than 3 per cent. of ash, containing a very small quantity (0.02 per cent.) of sand. The sand was estimated by extracting the ash with boiling 10 per cent. hydrochloric acid. A specimen of ochre tested by the author yielded 10 per cent. of soluble matters to 10 per cent. hydrochloric acid, and mixtures of this ochre with genuine ground cinnamon had properties similar to the samples in question. W. P. S.

Indian Kapok Seed. (*Bull. Imperial Inst.*, 1920, **18**, 335-337.)—The seeds of Indian kapok (*Bombax malabaricum*, D.C.), a large deciduous tree occurring throughout India and Ceylon, contained 8.9 per cent. of moisture and 22.3 per cent. of oil. The oil was bright yellow in colour, deposited some stearine on standing, and had the following characters: Sp. gr. at 15°/15°, 0.9208; $[\eta]_{15}^{20}$, 1.461; solidif. pt. of fatty acids, 38.0° C.; acid value, 9.3; saponification value, 193.3; iodine value, 78.0; soluble volatile acids, nil; and insoluble volatile acids, equivalent to 0.5 c.c. $\frac{N}{10}$ alkali per 5 grms. of oil. The oil therefore differs somewhat from that of Java kapok (*Eriodendron anfractuosum*), which has an iodine value 95 to 110. The composition of the residual meal was: Moisture, 11.4; crude proteins, 36.5; fat, 0.8; carbohydrates, 24.7; fibre, 19.9; and ash, 6.7 per cent. The cake obtainable from the seeds of Indian kapok and pressed to an oil content of about 7 per cent. would therefore contain about 34 per cent. of proteins in comparison with 25 to 26 per cent. in commercial undecorticated cotton-seed or Java kapok-seed cake. R. G. P.

Estimation of Coconut and Palm Kernel Oils in Fat Mixtures in which both Oils may be present. W. N. Stokoe. (*J. Soc. Chem. Ind.*, 1921, **40**, 57-58 T.)—The methods given by Burnett and Revis (*ANALYST*, 1913, **38**, 255) and by Blichfeldt (*ANALYST*, 1919, **44**, 290) having proved somewhat unsatisfactory, the author has devised the following method for estimating coconut and palm kernel oils

in mixtures. In the standard Reichert-Polenske process, after 110 c.c. have been distilled, the liquid is cooled and the insoluble acids collected on a filter; the condenser tube is rinsed down with 20 c.c. of water at 30° to 40° C., this being poured over the filter. The filter paper is washed with water at the same temperature, and as soon as the last drop of water has drained through, several capillary tubes of 1 mm. bore are filled to a depth of half an inch with the now melted acids. Two of the tubes are immediately attached, by means of a small rubber band, to either side of a thermometer graduated to read to 0.1° C., and the thermometer then fitted through the cork of a test-tube. The tube is supported in a beaker containing ether, the surface of which should be above the level of the acids in the capillary tubes. Whether the bulk of the acids is from palm kernel or from coconut oil will have been ascertained previously from the appearance of the acids in the original Reichert-Polenske distillate; if the acids are solid at the ordinary temperature it is necessary to warm the ether to about 30° C. A gentle stream of air is forced, by means of a foot-bellows, through the ether, so that the temperature of the latter is gradually lowered, the rate of cooling being adjusted to make the temperature of the ether 2° C. below that indicated by the thermometer within the test-tube. As the cooling progresses, the fatty acids become slightly cloudy, a distinct "seeding" or crystallisation afterwards occurring; at the first appearance of crystals the temperature is noted. This "seeding point" is quite definite and sharp, the acids in the capillary tubes appearing liquid, with a number of tiny white crystals along the sides of the tube; regular cooling is, however, essential.

The maximum, minimum, and average values obtained for the seeding point of four samples of coconut oil acids were 11.4°, 9.9°, and 10.75° C., whilst for four samples of palm kernel acids the corresponding values were 23.2°, 22.05°, and 22.75° C. For mixtures of acids of the two kinds, the seeding points, when plotted against the compositions, lie on a curve which is slightly concave towards the axis of temperature. The presence of other fats raises the seeding point somewhat, but the curves are approximately parallel to the original one. Thus, in the analysis of an unknown fat mixture, it is necessary first to ascertain the approximate proportion of the fats of the coconut group present by determining the saponification value and the Reichert-Polenske value, the relative proportions of coconut and palm kernel oils being read off from the corresponding setting point curve. The contents of the capillary tubes may be subsequently added to the alcoholic solution of the remainder of the insoluble acids, and the whole titrated as usual to obtain the Polenske value. T. H. P.

Composition of the Head Oil of the Sperm Whale. M. Tsujimoto. (*J. Chem. Ind., Japan*, 1920, 24, No. 275 Reprint.)—A specimen of crude oil obtained from the head of a male sperm whale (*Physeter macrocephalus*, L.) was cooled at 6° C. and pressed through a bag to remove spermaceti. The expressed oil had a light yellow colour, an unpleasant odour, and the following physical and chemical characters: Sp. gr. at 15°/4° C., 0.8848; $[\eta]_{15}^{20}$, 1.4633; acid value, 0.99; saponification value, 147.1; iodine value (Wijs), 71.4; Reichert-Meissl value, 0.57; unsaponifiable matter, 36 per cent.; fatty acids, 65 per cent.; glycerol, 3.52 per cent.; cholesterol, 0.18 per cent. The unsaponifiable matter (wax alcohols)

consisted chiefly of cetyl alcohol and oleicalcohol together with a small amount of pentadecyl alcohol. The fatty acids consisted of about 19 per cent. of solid acids (including palmitic and myristic acids) and about 81 per cent. of liquid acids (mainly physetoleic and oleic acids). On hydrogenation, the physetoleic acid yielded an iso-palmitic acid, m.-pt. 55° to 56° C.

W. P. S.

Fatty Oils from Reptilia. M. Tsujimoto and S. Kobayashi. (*J. Chem. Ind., Japan*, 1920, 23, No. 273 Reprint.)—Oils obtained from a Japanese viper (*Agkistrodon blomhoffii*), python (*Python reticulatus*), toad (*Bufo japonicus*), and giant lizard (*Varanus*), had the following characters :

	Viper Oil.	Python Oil.	Lizard Oil.	Toad Oil.
Specific gravity at 15°/4° C. ...	0.9192	0.9165	0.9132 (40° C.)	0.9348
Refractive index at 20° C. ...	1.4723	1.4685	1.4747 (40° C.)	1.4742
Acid value	0.4	0.6	21.0	8.5
Saponification value	187.7	194.1	188.8	181.3
Iodine value (Wijs)	110.8	80.3	101.0	104.4
Unaponifiable matter, per cent. ..	1.98	—	—	7.94
Ether-insoluble bromides, per cent.	7.8	2.1	9.7	1.0

W. P. S.

Test for Annatto in Fats and Oils. W. Brinsmaid. (*J. Ind. Eng. Chem.* 1921, 13, 216-217.)—The usual test for annatto, in which the fat is treated with sodium hydroxide solution and filtered, the dried filter-paper being subsequently tested with a drop of stannous chloride solution, is unsatisfactory, since the paper is frequently so greasy that the reagent does not come into contact with the annatto. The following modification of the method overcomes this difficulty. Fifteen c.c. of the clear melted fat are mixed thoroughly with 15 c.c. of chloroform and 15 c.c. of 5 per cent. sodium hydroxide solution, and the mixture is heated at 55° C. until the emulsion is broken up; the soap froth is then transferred to a beaker, 10 c.c. of water and 2 c.c. of sodium hydroxide solution and a suitable quantity of filter-paper pulp are added, and the mixture is heated on a steam-bath for thirty minutes. The filter-paper pulp is then collected by filtering the mixture through a Gooch crucible containing a disc of filter-paper, slight suction being applied. If annatto be present, the pulp will exhibit an orange colour, which changes to pink on the addition of a few drops of stannous chloride solution.

W. P. S.

Estimation of Maltose or Lactose in Presence of other Reducing Sugars: Use of Barfoed's Solution. Legrand. (*Comptes rend.*, 1921, 172, 602-604.)—Since Barfoed's solution is reduced by monoses but not by bioses, the proportion of maltose or lactose in a saccharine solution may be estimated by difference from the separate reductions of Fehling's and Barfoed's solutions. Fifteen c.c. of the latter

solution and 5 c.c. of sugar solution containing at most 0.1 grm. of sugar are boiled for three minutes in a conical flask; the slight loss of acetic acid occurring under these conditions does not involve formation of basic copper salt. The cuprous oxide separating is collected on an ordinary filter and estimated, according to Bertrand's method, by means of ferric sulphate and permanganate. This method is the only one which is applicable to the estimation of the amount of sucrose converted into invert sugar in condensed milk.

T. H. P.

Cattle Food prepared from Hydrolysed Sawdust. E. C. Sherrard and G. W. Blanco. (*J. Ind. Eng. Chem.*, 1921, 13, 61-65.)—Comparison of the analysis of the original wood with that of the completed stock food obtained by hydrolysis shows a reduction in the pentosan yield from 9.40-9.86 per cent. to 4.38-4.66 per cent., the loss being probably due to partial conversion of the pentoses into volatile acids and furfural. Methyl pentosans in the wood are unaffected by the sulphuric acid treatment; the amounts in the original wood were 2.37 and 2.83, and in the hydrolysed product, 2.15 and 2.50 per cent. The yield of cellulose from sawdust was 55.79 per cent., and from the cattle food 37.08 per cent., equivalent to 34.11 per cent. if calculated on the original woods; and the yield of reducing sugars was 71.5 per cent. of the theoretical figure. Treatment of cellulose from sawdust with 17.5 per cent. sodium hydroxide left over 50 per cent. unattacked, the amounts of α -cellulose in two samples being 57.30 and 55.85 per cent. Cellulose from hydrolysed wood was converted by alkali treatment into a viscous, semi-transparent mass, having the appearance of collodion.

W. J. W.

Estimation of the Alkalinity and Phosphate Content of the Ash of Foods. J. Tillmans and A. Bohrmann. (*Zeitsch. Unters. Nahr. Genussm.*, 1921, 41, 1-16.)—Alkalinity due to oxides and carbonates is estimated by treating a portion of the ash with an excess of standard acid, boiling the mixture to expel carbon dioxide, adding 30 c.c. of 40 per cent. calcium chloride solution, and titrating the excess of acid with standard alkali solution, using phenolphthalein as indicator. The addition of the calcium chloride prevents the interference of the phosphates present. To estimate phosphates, another portion of the ash (at least 0.2 grm.) is boiled for one hour with 100 c.c. of $\frac{N}{10}$ hydrochloric acid, cooled, and titrated with $\frac{N}{10}$ sodium hydroxide solution, using methyl-orange as indicator; let the quantity of $\frac{N}{10}$ acid used = b . Twenty c.c. of saturated sodium oxalate solution are then added and the solution is titrated until a faint pink coloration with phenolphthalein is obtained; let the quantity of standard solution used for this titration = c . If the ash be alkaline (equal to a c.c. of $\frac{N}{10}$ acid solution), then orthophosphates only can be present, and the formula $\frac{3}{2}(b-a) \times 3.167$ gives mgrms. of PO_4 present as orthophosphate. When the ash is not alkaline and the value c is positive, the orthophosphate $-\text{PO}_4 = 3c \times 3.167$ and the pyrophosphate $-\text{PO}_4 = 2(b-2c) \times 4.75$ mgrms. If c has a negative value, then pyro- and metaphosphates are present; in this case the pyrophosphate $-\text{PO}_4 = 2b \times 4.75$ and the metaphosphate $-\text{PO}_4 = -c \times 9.5$ mgrms. Milk ash contains a small quantity of alkali as oxides and carbonates, and 30 to 40 per cent. of orthophosphates; pyro- and metaphosphates are not present. Flour ash contains no true alkali, but a

mixture of the three phosphates. Beef and horse-flesh ashes contain no oxide or carbonate, but about 7 per cent. of orthophosphate and 50 per cent. of pyrophosphate. Fruit juice ashes consist chiefly of carbonates. Cocoa ash has a small alkalinity due to oxides and carbonates; it contains 33 per cent. of orthophosphates.

W. P. S.

The United States Pharmacopœia Method for the Estimation of Phosphoric Acid and Soluble Phosphates. A. E. Stearn, H. V. Farr and N. P. Knowlton. (*J. Ind. Eng. Chem.*, 1921, 13, 220-225.)—The fairly accurate results yielded by the method prescribed in the U.S. Pharmacopœia are obtained only at the specific concentration given, namely, 0.0062 gr. per c.c.; at other concentrations the results obtained are untrustworthy. This is probably due to the formation of silver hydrogen phosphates which are slightly soluble, the amount increasing rapidly as the phosphate concentration is increased and the excess of silver nitrate simultaneously decreased. If the method be modified by converting the acid into the trisodium salt, the results obtained are trustworthy and are independent of the concentration. Ten c.c. of a 1 per cent. solution of the acid are treated with 5 grms. of sodium nitrate, cooled to 0° C., and titrated with $\frac{N}{10}$ sodium hydroxide solution, using phenolphthalein as indicator; the number of c.c. of alkali solution required is divided by two, and this quantity is added in excess. The mixture is then treated with 50 c.c. of $\frac{N}{10}$ silver nitrate solution, diluted to 100 c.c., mixed, filtered, and 50 c.c. of the filtrate are acidified with nitric acid and the excess of silver titrated with thiocyanate solution. The modified method may be applied to sodium phosphates; in this case the salts are first rendered anhydrous and the amount of alkali required is found by calculation.

W. P. S.

Estimation of Santonin in Wormseeds. T. Kariyone and Y. Kimura. *Pharm. Soc. Japan*, 1920, 927-940; *J. Chem. Soc.*, 1921, 120, ii., 223.)—The method is essentially a determination of the saponification value, as a measure of lactone formation. The residue left after a Soxhlet extraction of 10 grms. of powdered *Artemisia Cina*, Berg., with ether, is boiled with 100 c.c. of 5 per cent. barium hydroxide solution, acidified with dry carbon dioxide and filtered. Eighty c.c. of the filtrate are extracted with 20 c.c. of chloroform and 10 c.c. of 15 per cent. hydrochloric acid, and then with three portions of 10 c.c. of chloroform. The extract is evaporated, the residue dissolved in 30 c.c. of alcohol, and the solution neutralised with $\frac{N}{10}$ potassium hydroxide solution, 20 c.c. of which are then added in excess. The mixture is heated under a reflux condenser for half an hour and titrated with $\frac{N}{10}$ hydrochloric acid, using phenolphthalein. When S' and S are the amounts of $\frac{N}{10}$ acid required by the 20 c.c. of potassium hydroxide and the sample respectively, the santonin content per cent. is expressed by the formula $2.462 (S' - S)/8$.

H. E. C.

The Alkaloid and Oil Content of Meadow Saffron Seeds. C. Grimme. (*Chem. Abstracts*, 1921, 15, 574; from *Pharm. Zentralhalle*, 1920, 61, 521-4.)—The content of colchicine varied from 0.11 to 0.52 per cent., the actual amount present

being from 1 to 25 per cent, greater than indicated by the physiological activity. The proportion of alkaloid present was higher in the smaller seeds. The seeds yielded 6.6 to 8.4 per cent. of an oil having the following characteristics: Sp. gr., 0.9176 at 15° C.; solidifying-point, -9° C.; $[n]_D^{20}$, 1.4642; acid value, 20.3; saponification value, 184.3; iodine value, 128.5; and unsaponifiable matter, 0.71 per cent. The fatty acids had: M.-pt., 24° C.; solidifying-point, 22.5° C.; $[n]_D^{20}$, 1.4646; neutralisation value, 187.6; and iodine value, 131.0.

T. J. W.

Clove Oil from Clove Stems. S. T. Gadre. (*J. Indian Ind. and Labour*, 1921, 1, 41-47.)—The oil was prepared by soaking the stems connecting the flowers to the branches in water overnight, and distilling with steam at a pressure of from 10 to 15 lbs. for five or six hours in a tin-lined copper still. A yield of 4.50 per cent. of a heavy, pale-yellow oil was obtained, equal in aroma and quality to the best English distilled clove oil. Examination of the oil gave the following results: Sp. gr., 1.0522 at 34° C.; $[n]_D$, 1.5345; total eugenol content (Umney's method), 93.09 per cent. (Thom's gravimetric method), 83.53 per cent.; free eugenol content by Karley and Bolsing's volumetric method, 69.86 per cent. The benzoyl-eugenol obtained in Thom's methods on recrystallisation from alcohol gave a m.-pt. of 70° C. The oil was soluble in half its volume of 80 per cent. alcohol, but with more than two volumes an opalescence was produced.

T. J. W.

Essential Oil of Spearmint from South Africa. (*Bull. Imperial Inst.*, 1920, 18, 350-351.)—Dried leaves of spearmint (*Mentha longifolia*) grown in the Cape Province yielded 2.4 per cent. of essential oil; the stems were practically devoid of oil. The essential oil showed the following characters: Sp. gr. at 15°/15°, 0.947; $[\alpha]_D = -47.6^\circ$; $[n]_D^{20}$, 1.4925; ketones (largely or entirely carvone), 70 per cent. The oil was similar in character to, but richer in ketones than, English and American spearmint oils (derived from *M. spicata*, Huds., or *M. viridis*, L.), which contain 30 to 48 and 35 to 66 per cent. respectively of ketones, and resembled Austrian spearmint oil, which contains up to 72 per cent. of ketones.

R. G. P.

Reaction of Saccharin. (A Correction.) L. Thevenon. (*J. Pharm. Chim.*, 1921, 23, 215.)—In dilute acid solution, β -naphthol reacts with nitrous acid and yields a red colouring matter. Consequently, the test for saccharin described recently (*ANALYST*, 1921, 54) is not characteristic of this substance.

W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Vitamins and the Food Supply. A. Harden. (*J. Soc. Chem. Ind.*, 1921, 40, 79-82 R.)—The vitamin requirements of the animal organism, the variations in the vitamin content of foodstuffs, and the influence of processes of manufacture or preparation, are considered. The three vitamins, fat-soluble A, water-soluble B or

* No temperature given.

anti-neuritic, and water-soluble *C* or anti-scorbutic, are termed, in accordance with Drummond's suggestion, vitamins *A*, *B*, and *C* respectively, and occur chiefly in (*A*) animal fats and products containing them, fish liver oils, green vegetables, and egg-yolk; (*B*) seeds (especially in the germ, pericarp, and aleurone layer), yeast, and egg-yolk; and (*C*) green vegetables (especially *Cruciferae*), orange and lemon juice, tomatoes, germinated seeds, swedes, and turnips; they cannot be synthesised by the mammalian organism, and are required in relatively large proportions by the young growing organism. The amount of vitamin contained in natural foodstuffs appears to vary under different conditions of season, age, etc., and may also be modified by the treatment to which the material is subjected prior to consumption. Thus, the unbalanced use of milled rice is the main cause of beri-beri in the tropics. The effect produced by drying varies very greatly with the character of the foodstuff. Cabbage, the richest known source of vitamin *C*, loses 70 to 80 per cent. of its anti-scorbutic potency when dried, even at a low temperature, whilst orange and lemon juices may be evaporated *in vacuo* with little or no loss. If undue exposure to a high temperature be avoided, dried milk may be prepared but little inferior in anti-scorbutic properties to fresh milk, although many samples of dried milk actually on the market contain far less of the antiscorbutic principle than fresh milk. Vitamins *A* and *B* are less affected by desiccation than vitamin *C*, although with these also the effect varies with the nature of the foodstuff. Comparatively little is known of the effect of preservation on the vitamin content of food materials. Air-dried cabbage retains its slight anti-scorbutic potency unimpaired for long periods when preserved over phosphorus pentoxide, even at 37° C., but loses it rapidly when kept over calcium chloride. It seems probable that a perfectly dry powder will long withstand preservation at ordinary temperatures, whereas in the presence of even very small amounts of moisture deterioration may occur. The deterioration of lemon juice in this respect is prevented by the natural antiseptic furnished by the oil of the rind. Vitamin *A* is moderately stable towards rise of temperature provided that air is excluded, whereas in the presence of air it becomes rapidly inactivated, even at comparatively low temperatures; it remains almost unaltered when cabbage is cooked or when milk is autoclaved at 120° C. for an hour. Vitamin *B* is but little affected by heating for one to two hours at 100° C., and has even been found to survive boiling with sulphuric acid. As regards vitamin *C*, matters are less definite, the effect of heat in this case appearing to depend largely on the nature of the vitamin-containing material.

In the course of refining fish oils seem to lose much of their vitamin *A*, which is absolutely destroyed by hydrogenation—either by the actual reduction, or by the catalyst or the conditions employed.

T. H. P.

Estimation of a Substance from Yeast and Rice Polishings which Accelerates Fermentation. S. Fränkel and E. Schwarz. (*Biochem. Zeitsch.*, 1920, 112, 203-235; *J. Chem. Soc.*, 1921, 120, ii., 228.)—The fermentation-accelerating factor from yeast and rice polishings can be estimated as follows: The fats are removed from an 80 per cent. alcohol extract of yeast, which is then treated with basic lead acetate. Excess of lead is removed from the

filtrate by hydrogen sulphide, and mercuric chloride added. After decomposition of the precipitate with hydrogen sulphide, it is freed from hydrochloric acid and concentrated in a vacuum. An inactive picrotonate is precipitated by adding picrotonic acid, and the active substance precipitated with phosphotungstic acid. This precipitate is decomposed with baryta and sulphuric acid and concentrated in a vacuum, yielding an active base twenty-two times as strong as the original alcoholic extract.

H. E. C.

Amylase of *Rhizopus Tritici*. L. L. Harter. (*J. Agric. Research*, 1921, 20, 761-786.)—The mould was cultivated on a modified Czapek's solution with varying amounts of starch paste, glucose, or both, at 25° to 35° C., for seven to ten days. The felt of mycelium was washed with water, acetone, and ether, dried by exposure to air, and kept at 9° C. until required. The results of a large number of experiments are tabulated, the following results being deduced: an energetic amylase is produced during the growth of the mould, a proportion of which diffuses out into the culture solution. The enzyme exerts a vigorous action upon starch paste, but raw potato starch is attacked less readily, the optimum temperature being 45° C., whilst at 60° C. the amylase is completely destroyed in 100 hours. The addition of glucose causes retardation in the hydrolysis of starch by the amylase, but has no other influence upon the results. Since the determination of the reducing sugars does not account for the whole of the starch when an end point is shown by iodine, it is probable that dextrins are formed during the hydrolysis. Mycelium grown at 9° C. hydrolyses four times as much starch as that grown at 40° C., whilst cultivation at 29° C. yields an intermediate figure, although the fungus grows best at the latter temperature. The activity of the enzyme depends upon the influence which the carbohydrate added to the culture medium has upon the growth of the fungus, the most efficient source of carbohydrate being sweet potato bouillon.

T. J. W.

Composition of Tubers, Skins, and Sprouts of Three Varieties of Potatoes. F. C. Cook. (*J. Agric. Research*, 1921, 20, 623-635.)—Specimens of Rural New Yorker, Green Mountain, and Irish Cobbler varieties of potatoes were stored in a dark chamber at 70° C. for four to eight months, and the sprouts, skins, and tubers separated and analysed. Extensive details are given of the results obtained, for which the original paper should be consulted. The composition is practically identical for the three varieties, and spraying with Bordeaux mixture caused no variation in composition. The sprouts contained more protein and less basic nitrogen than the skins and tubers, and contained a larger proportion of water, ash, phosphoric acid, and nitrogen than originally present in the tuber. Copper was found in both sprayed and unsprayed samples, varying from fourteen to forty-one parts per million.

T. J. W.

Digestion of Vegetable Fibres. W. Thomann. (*Chem. Zeit.*, 1921, 45, 200.)—Digestion with ammoniacal copper oxide, as suggested by Mach and Lederle, gives results in close agreement with those obtained by the agency of ruminating animals. In the case of rabbits digestion is less complete, and values obtained by

Mach's method must be multiplied by 0.49 and 0.72 for straw fodder and grain food respectively.
W. J. W.

Hemicellulose of Apple Wood. W. E. Tottingham, R. H. Roberts, and S. Lepkousky. (*J. Biol. Chem.*, 1921, 45, 407-414.)—The base wood of young fruiting apple shoots was analysed by drying, extracting with ether and 90 per cent. alcohol, digesting with saliva, and boiling the residue with 2.5 per cent. (volume) sulphuric acid for two hours under a reflux condenser. The amount of material (hemicellulose) hydrolysed by this treatment amounted to 28.7 per cent., calculated on the original dry wood. The acid extract, after neutralising, decolorising, and purifying, yielded a very viscous pale brown solid, which on examination by fractional precipitation of osazones and quantitative tests proved to be a mixture of dextrose, xylose, and a small proportion of galactose, together with other unidentified substances. It is suggested that the hemicellulose forms a carbohydrate reserve in the metabolism of the apple-tree.
T. J. W.

Detection of Albumoses. C. Achard and E. Feuillee. (*J. Pharm. Chim.*, 1921, 23, 146-147.)—Albumin is first removed from the liquid (*e.g.*, blood-serum) as follows: 2.5 c.c. of the liquid are diluted with 10 c.c. of water in a 100 c.c. conical flask, 1 drop of acetic acid and 1.25 gm. of sodium chloride are added, and the flask heated over a wire gauze, with constant rotation and removal from the flame whenever the foam rises in the flask. After two to four minutes' boiling there is incipient coagulation, and at this point the liquid is allowed to cool for about three minutes, again heated, and immediately it boils is filtered into a tube 2 cm. in diameter, and made up to 10 c.c. with boiling water and filtered. The filtrate is treated with 10 c.c. of Tanret's reagent and again heated, care being taken that on removing the tube from the flame directly ebullition occurs the liquid is clear. After cooling the tube in a stream of water, the turbidity due to albumoses appears, and is compared, after standing for one hour, with standard tubes of Witte's peptone.
R. G. P.

Estimation of Amino-Acids in Urine. W. Mestrézal. (*J. Pharm. Chim.*, 1921, 23, 137-141.)—The following modification of Sörenson's method obviates the use of litmus as an indicator. Phosphates and carbonates are first removed as follows: To 50 c.c. of urine in a 100 c.c. graduated flask 2 grms. of powdered barium chloride and two drops of phenolphthalein solution are added, and when the barium chloride is dissolved, saturated barium hydroxide solution is added (5 c.c. in excess of that required to colour the phenolphthalein), and the liquid is allowed to stand in the corked flask for fifteen minutes and then made up to 100 c.c. After filtration, 20 c.c. of liquid (= 10 c.c. of urine) are treated with ten drops of phenolphthalein solution (1 per cent.) and decolourised by adding dilute hydrochloric acid drop by drop; $\frac{N}{10}$ sodium hydroxide solution is then run in till a pale rose tint appears. Ten c.c. of neutral formaldehyde solution (35 to 40 per cent.) are next added, and the liquid is finally titrated with $\frac{N}{10}$ sodium hydroxide solution, the titration being continued until the decided red tint of the blank is reached. The blank (or standard

tint) is obtained by taking 10 c.c. of neutral formaldehyde solution, 20 c.c. to 30 c.c. of boiled distilled water, 1 c.c. of phenolphthalein, and adding $\frac{N}{10}$ sodium hydroxide (0.3 c.c. to 0.4 c.c.) until a decided red tint is obtained. It is necessary to continue the titration to this point, as the amino-acids react with formalin to produce derivatives (of the type $R-CH(N=CH_2)COOH$) which function as weak acids. Allowance is made for the retarding effect of ammonium salts by increasing the number of c.c. taken for titration by $\frac{1}{10}$. The amino-nitrogen is obtained by deducting the ammoniacal nitrogen determined separately by Schloesing's method.

R. G. P.

WATER ANALYSIS.

Analysis of Mineral Sulphide Waters. J. G. Fairchild. (*J. Washington Acad. Sci.*, 1920, 10, 559-565; *J. Chem. Soc.*, 1921, 120, ii, 126.)—In waters containing alkali and alkaline earth hydrosulphides and hydrogen carbonates the alkalinity increases with the escape of hydrogen sulphide or precipitation of sulphur. Calcium and magnesium chlorides are pronouncedly acidic towards the alkali sulphides, but have less effect on the hydrogen carbonates. Barium chloride aids in the decomposition of hydrogen carbonate ions in carbonate waters, but has no such effect where much alkali sulphide is present. It is proposed to estimate carbon dioxide present as hydrogen carbonate, and volatile hydrogen sulphide, by boiling the water rapidly for five minutes in a current of hydrogen, and absorbing the gases in an ammoniacal solution of cadmium and barium chlorides; on acidification of this solution with acetic acid the carbon dioxide is liberated and collected, and the cadmium sulphide remains.

R. G. P.

The Effect of Denitrifying Bacteria in Water. K. Scheringa. (*Pharm. Weekblad*, 1921, 58, 263-269.)—A culture of *B. pyocyaneus*, added to waters from various supplies, in no case exerted a denitrifying action; such action may in fact even be impeded by these bacteria, and nitrites are usually formed. With only a small content of organic matter, little change occurs; in presence of an appreciable amount, however, loss of nitrogen compounds results when samples of water are kept at moderately high temperatures in absence of air.

W. J. W.

AGRICULTURAL ANALYSIS.

Colloid Chemistry of Indicators. G. Wiegner. (*Chem. Zeit.*, 1921, 45, 200.)—The use of azolitmin as an indicator in soil analysis is based on the blue coloration which it gives with calcium ions; addition of potassium chloride increases the sensitiveness of the indicator owing to formation of calcium chloride. The retarding effect of humus is overcome in presence of excess of calcium ions.

W. J. W.

ORGANIC ANALYSIS.

Volumetric Method for the Estimation of Total Sulphurous Acid in Organic Substances. V. Froboese. (*Arbeit. Reichsgesundheitsamte*, 1920, 52, 657-669.)—A quantity of the substance, containing not more than 90 mgrms. of sulphur

dioxide, is placed in a flask, together with 400 c.c. of water and 50 c.c. of 25 per cent. phosphoric acid; a current of carbon dioxide is passed into the flask, which is attached to a condenser, and the liquid is distilled, the distillate being collected in a receiver containing 40 c.c. of $\frac{N}{10}$ sodium hydrogen carbonate solution. When 200 c.c. of distillate have been collected, an excess of hydrogen peroxide is added, the mixture boiled until oxygen and carbon dioxide are no longer evolved, and the excess of alkali (carbonate) is titrated with $\frac{N}{10}$ hydrochloric acid, using methyl-orange as indicator. Each c.c. of $\frac{N}{10}$ sodium bicarbonate is equivalent to 3.2 mgrms. of sulphur dioxide. If the substance under examination contains volatile acids other than sulphurous acid, the distillation flask should be connected with the condenser by means of a long vertical tube; this arrangement is usually effective in preventing the distillation of the acids. The use of a current of carbon dioxide in the distillation is simply to aid the distillation of the sulphurous acid; it does not prevent the oxidation of the latter if any dissolved oxygen is present in the water.

W. P. S.

Estimation of Small Quantities of Iron in Organic Liquids, especially in Wines. Malvezin and C. Rivilland. (*Ann. Chim. anal.*, 1921, 3, 90-92.)—The method depends on the titration of ferric salts with thiosulphate solution, using sodium salicylate as indicator. The ash obtained from 20 c.c. of wine is dissolved in 10 c.c. of 5 per cent. hydrochloric acid, the solution filtered, and the filtrate treated with 0.5 c.c. of hydrogen peroxide. After fifteen minutes the solution is boiled to decompose the excess of peroxide, 5 c.c. of 1 per cent. copper sulphate solution and 1 c.c. of 2 per cent. sodium salicylate solution are added, and the mixture is titrated with standardised (about 0.5 per cent.) sodium thiosulphate solution until the violet coloration is discharged. The copper sulphate acts as a catalyst and aids the reduction of the ferric salt.

W. P. S.

A Method of Purification of Methyl Alcohol. A. Lanzenberg and J. Duclaux. (*Bull. Soc. Chim.*, 1921, 29, 135-136.)—A method of purification of methyl alcohol, especially from ethers and ketones of near boiling-points, is based upon the formation by methyl alcohol of a eutectic mixture of b.-pt. 50° C., whereas acetone, for example, forms an anti-eutectic mixture boiling 12° C. higher and easy to separate. One part of the crude alcohol is mixed with 7.5 parts (by weight) of chloroform and distilled in a rectifier, the fraction boiling between 52.5° and 53.5° C. is collected, acetone-chloroform mixtures and other impurities coming over above 64° C. This distillate is washed three times with an equal volume of water and the aqueous extracts are distilled. A second distillation will yield a practically anhydrous methyl alcohol. This product may contain about 0.25 gm. acetone per 100 c.c. as sole impurity.

H. E. C.

American Turpentine. (*U.S. Dept. Agric. Bureau Chem. Bull.* 898, 43-49.)—The following specifications are recommended by the U.S. Interdepartmental Committee on Paint Specification Standardisation (October, 1919) for turpentine and wood-turpentine—i.e., turpentine distilled from resinous wood. The turpentine

shall be clear and free from suspended matter and water, its colour shall be "standard" or better, and it shall possess the characteristic odour of the variety of turpentine specified, or conform to that of an agreed sample; sp. gr. at 15.5°/15.5° C., 0.862 to 0.875; $[n]_D^{20}$, 1.468 to 1.478; initial boiling-point at 760 mm., 150° to 160° C.; fraction below 170° C. at 760 mm., at least 90 per cent. by volume; polymerisation test for turpentine (derived from oleo-resin), maximum residue 2.0 per cent. by volume with $[n]_D$ at 20° C. above 1.500; polymerisation test for wood-turpentine, maximum residue 2.5 per cent. with $[n]_D^{20}$ above 1.495.

Full details are given of methods of sampling and analysis. The determination of initial boiling-point and per cent. of distillate below 170° C. is carried out under standard conditions in an apparatus adopted by the American Society for Testing Materials for paint thinners, of which a dimensioned drawing is given. The polymerisation test is carried out by treating the turpentine (5 c.c.) with 38 N-sulphuric acid (20 c.c.) in a Babcock bottle; the turpentine is mixed with the cold acid, keeping the temperature below 60° C., until the mixture no longer warms up on shaking; the flask is then heated for not less than ten minutes to 60° to 65° C. on the water-bath, and shaken six times for half-a-minute each time (the stopper should not be used, as the flask may burst). After cooling to room temperature, sulphuric acid is added until the unpolymerised residue reaches the graduated neck and the volume of residue is read after separation by standing for twelve hours or by centrifuging. The residue should be viscous and straw-coloured, or darker. A comparative blank test should be made with turpentine of known purity. Colour is determined by matching in a tube graduated in millimetres. The following grades are recognised by the depth of column required to match a No. 1 yellow Lovibond tintometer glass: water white, 150 mm.; standard, 50 mm.; one shade off, 25 mm.; two shades off, 15 mm.

R. G. P.

Syntheses of Cyanic Acid by Oxidation of Organic Substances. New Methods of Analysis of this Substance. R. Fosse. (*Bull. Soc. Chim.*, 1921, 29, 158-203.)—Urea is an excretion product of vegetable as well as animal life, and results from the oxidation of both proteins, carbohydrates, and fats. Cyanic acid is the intermediate product. Urea is precipitated quantitatively in the form of crystals of urea dixanthylate by xanthidol in methyl-alcohol solution in presence of acetic acid; cyanic acid may be similarly estimated by its hydrolysis by acid or alkalies to urea and precipitation with xanthidol, which gives accurate results even with minute quantities. In the case of mixtures of cyanic acid and urea, two determinations of urea by xanthidol are made, one direct and one after heating an hour with ammonium chloride; the difference is a measure of the cyanic acid. This acid may also be estimated by means of its silver salt; ammonium chloride and silver cyanate with water are heated for fifteen minutes on the water-bath, a little acetic acid and xanthidol added, and the crystals separated by centrifuging; 0.5 mgrm. of cyanic acid may be so treated. Nitric acid interferes with the reaction. Cyanic acid may be identified microchemically by the characteristic filamentous crystals of the silver salt, which gives a blue colour with cobalt acetate. On shaking with water and amyl alcohol this is not transferred to the latter as in the case of a

sulphocyanate. Silver cyanate may be analysed quantitatively by heating it with ammonium chloride and weighing the resulting silver chloride, and estimating the urea as dioxanthylate. H. E. C.

Characters of Resins used for making Varnishes. P. Nicolardot and C. Coffignier. (*Chim. et Ind.*, 1921, 5, 150-156.)—To determine the relative hardness of different resins the authors employ an apparatus which consists of a vertical rod fitted with a steel ball at its lower end; the block of resin is supported below this rod, so that the ball rests on the surface, and weights are applied to the upper end of the rod. Usually the ball has a diameter of 2 mm., and the weight used is 5 kilos. After the ball has been pressed on the resin for varying periods—say, five seconds, ten seconds, and ten minutes—the diameter of the impression produced is measured by means of a microscope. During the test the resin is maintained at a definite temperature (0° C. or 25° C.). The hardness and other characters of resins are given as follows, the values for the hardness being the diameter of the impressions in millimetres, magnified by 44.5:

	Specific Gravity.	Melting-Point.	Hardness—		
			5 Seconds.	10 Seconds.	10 Minutes.
C.					
Hard resins :					
Zanzibar	1.058	> 300	22	—	23
Madagascar	1.056	> 300	23	—	23
Demerara	1.047	180	23.5	—	23
Semi-hard resins :					
Congo	1.061	195	23	23.5	—
Benguela	1.058	165	24	24.5	—
Cameroon	1.052	150	23	24.5	—
Angola, red	1.066	> 300	22	25	—
Kissel	1.066	110	22	25	—
Brazil	1.053	100	23	25	—
Angola, white	1.055	95	25	26	—
Sierra Leone	1.072	130	25	28	—
Soft resins :					
<i>Aucoumea Klaineana</i>	0.996	77	77	40.5	50
<i>Hopea odorata</i>	0.990	110	53	47	—
<i>Hopea dealbata</i>	1.061	142	82	72	—
Various :					
Amber, yellow	1.052	> 300	22	25	—
Amber, cloudy	1.052	> 300	29	29	—
Kauri, light	1.036	165	28	41	—
Kauri, bush	1.030	150	29	44	—
Kauri, crop	1.038	125	36	80	—
Manilla, hard	1.065	190	26	38	—
Manilla, soft	1.060	120	28	49	—
Pontianac	1.037	135	69	95	—

Assay of Coal for Carbonisation Purposes. T. Gray and J. G. King. (*Fuel Research Board, Technical Paper* No. 1, 1921.)—The authors have devised a method and apparatus for the measurement and analysis of the primary products from coal carbonisation, by which secondary changes are avoided. The apparatus comprises an electric furnace; a hard glass or silica tube sealed at one end and closed by a rubber stopper at the other, and having a short lateral branch; a U-shaped condensing tube with bottom stop-cock; an absorption tube containing glass beads soaked in sulphuric acid and also provided with a stop-cock; and a gas-holder filled with glycerin and water. This gas-holder is connected at the bottom by means of a rubber tube with a glass reservoir suspended over two pulleys, having a counterpoise floating in a vessel, into which further liquid can pass from the reservoir by means of an overflow tube; by this arrangement any desired pressure may be maintained in the gas-holder. The sample of coal is ground to pass a 60-mesh sieve and dried at 105° to 110° C.; 20 grms. are then introduced into the hard-glass tube, previously weighed, and the weighed U-tube is attached, the whole being then connected with the remainder of the apparatus. After the furnace has been heated to 300° C., the sample tube is introduced and the temperature is raised to 550° to 600° C., in one hour, observations being made of the temperatures at which water and oil appear; heating is continued for one hour. The oil and water in the U-tube, after weighing, are washed into a graduated measure with chloroform or petroleum ether, by which means the aqueous portion is determined. The yield of coke is obtained by weighing the cooled sample-tube, and ammonia and bases are estimated in the contents of the absorption flask and in the aqueous washings from the U-tube. To ascertain the gas volume, water is removed from the vessel in which the counterpoise floats and is allowed to run into the reservoir, which is then raised till the top of the inlet is level with the liquid in the gas-holder, and the liquid in the manometer tube is level. From the weight and specific gravity of the liquid in the counterpoise vessel, the gas volume at observed temperature and atmospheric pressure is calculated. In a second test the gas from the first test, which is to some extent contaminated by air, may be used to displace air from the apparatus.

W. J. W.

Hydrolytic Alkalinity of Pure and Commercial Soaps. F. C. Beedle and T. R. Bolam. (*J. Soc. Chem. Ind.*, 1921, **40**, 27-29t.)—In continuation of previous work the authors have determined the hydron concentration at 90° C. of aqueous solutions of different strengths of the sodium salts of oleic, palmitic and abietic acids, and of soaps of various types by the method of Francis and Geake (*ANALYST*, 1913, **38**, 522; 1916, **41**, 22; 1918, **43**, 358), depending on the decomposition of nitrosotriacetoneamine. Numerous results are given, and conclusions are drawn as to the hydrolytic alkalinity of different soaps and the irritant effect on the skin attributed to certain soaps—*e g.*, coconut oil soap.

R. G. P.

Estimation of Cellulose in Bast Fibres. Y. Uyeda. (*J. Ind. Eng. Chem.*, 1921, **13**, 141-143.)—In estimations of cellulose, Dore (*J. Ind. Eng. Chem.*, 1920, **12**, 264) found that Renker's method of chlorination without hydrolysis (*Bestimmungs-*

methoden der Cellulose, 1910) gives better results than either of the methods proposed by Cross and Bevan, or Johnsen and Hovey (*Paper*, 1918, 21, 36). An investigation of these methods has been applied to the estimation of cellulose in Korean hemp fibre, which was cut into pieces, 1 cm. long, for storage during the experiment. Preliminary treatment of the fibre consisted in drying 1 grm. samples for sixteen hours in an electric oven, and extracting them for six hours with benzene, and then for the same period with 95 per cent. alcohol. Here again, Renker's method was the most satisfactory, but the cellulose obtained by Cross and Bevan's method showed the highest degree of purity, as indicated by a determination of the α -cellulose by their mercerisation test; this test also shows the chemical behaviour of the fibre towards alkaline reagents. Pectin substances retained in the cellulose obtained by Renker's and Johnsen's methods are partly removed by the alkali in mercerisation. Pentosan and methylpentosan in bast fibres may be estimated by Tollen's and Kroeber's and Ellet and Tollen's (*J. Landw.*, 1905, 53, 20) methods respectively; but hexosans also yield oxymethylfurfural. Hemp fibres have in some respects a similar composition to that of wood cellulose, and their cellulose may have an oxycellulose structure, as its affinity towards basic dyestuffs is much greater than that of cotton. The cellulose, as well as the α -cellulose, from these fibres, on distillation with hydrochloric acid, gives a mixture of furfural, methylfurfural, and oxymethylfurfural; and a brown phloroglucide, consisting of the two last-named substances, was obtained from the α -cellulose. W. J. W.

Constitution of Cellobiose (Cellose). W. N. Haworth and E. L. Hirst. (*J. Chem. Soc.*, 1921, 119, 193-201.)—The structure of cellobiose has been investigated, and experimental evidence is brought forward in support of the constitutional formula proposed previously (*J. Chem. Soc.*, 1919, 115, 809). An improved method for the preparation of cellobiose consists in stirring for five minutes 20 grms. of dried filter-paper with 80 c.c. of commercial acetic anhydride (80 to 95 per cent.) containing 11 c.c. of sulphuric acid, and heating the paste on a bath at 120° C. until, at about 112° C., the mixture becomes a dark-red, mobile liquid and begins to boil; immediately the liquid appears to be changing to black it is poured into 1.5 litres of cold water. The pale yellow precipitate of cellobiose oct-acetate, which separates after about ten minutes (and which should be soluble in boiling, but not in cold, alcohol), is allowed to stand in contact with water for six hours, filtered, washed, and dried at 40° C., and then dissolved in boiling 90 per cent. alcohol, the solution filtered hot, and the octa-acetate crystallised (m.-pt., 224° to 227° C.; yield, 25 to 35 per cent. of cellulose taken). The acetyl groups are removed from the cellobiose oct-acetate by alcoholic potassium hydroxide, and the resultant potassium cellobiosate converted to cellobiose by treatment with perchloric acid. Cellobiose is hydrolysed by emulsin and by cellulase, with production of dextrose, but is not hydrolysed by maltase. It is not known whether the effect of emulsin is due to the presence of traces of cellulase. The optical properties of derivatives of lactose and of cellobiose are strikingly similar, and it is considered very probable that the linking of the two hexoses in each is structurally and stereochemically similar. Cellobiose may be regarded as glucose- β -glucoside. R. G. P.

Solubility of Crystalline Substances in Rubber. G. Brunl. (*Giorn. Chim. Ind. Appl.*, 1921, 3, 51-53.)—Thermal analysis of the binary systems formed by rubber with azobenzene, naphthalene, and *p*-toluidine shows that the rubber acts toward these crystalline compounds as an ordinary solvent, yielding true, saturated solutions, supercooling and supersaturation of these readily occurring. That complexes between the rubber and the crystalline components of the systems are formed is indicated by the pronounced concavity of the composition-temperature curves towards the axis of concentration. The molecular condition of the rubber is not altered by prolonged heating of the systems. T. H. P.

INORGANIC ANALYSIS.

Action of Iodine on Different Metals in the Cold: Process for the Detection of the Presence of Chlorine in the Atmosphere. C. Matignon. (*Comptes rend.*, 1921, 172, 532-534.)—If a small crystal of iodine be placed on a leaf of beaten silver, the latter undergoes conversion into friable silver iodide, the crystal becoming surrounded by a yellow circle, the radius of which increases with gradually diminishing rapidity. Calculation shows that a sheet of silver 3μ in thickness should be completely converted into iodide beneath the iodine in about a second. Other metals give somewhat similar results. This phenomenon may be employed to indicate the presence of chlorine in the air, the procedure being as follows: On a strip of silver leaf 1 cm. wide is placed a thin layer of moistened potassium iodide reaching from side to side of the strip, which is used to close an electric circuit. When chlorine comes into contact with the iodide, the iodine liberated converts the metal into non-conducting silver iodide and thus interrupts the current; if an ampèremeter be inserted in the circuit, the movement of the needle may serve to actuate an alarm. If the width of the rectangle of potassium iodide be 3 mm. and the silver leaf be 3μ in thickness and 1 cm. in width, 0.1 c.c. of chlorine will suffice to liberate enough iodine to convert the corresponding surface of the metal into iodide. Lead iodide or mercuric iodide may be used in place of potassium iodide, and bromine vapour acts appreciably more slowly than chlorine.

T. H. P.

Electrometric Titration of Hydriodic Acid and its Use as a Standard in Oxidimetry. W. S. Hendrixson. (*J. Amer. Chem. Soc.*, 1921, 43, 14-23.)—For estimating iodine ion in presence of other halides, a method of electrometric titration is used, in presence of an oxidising agent which can be standardised accurately and is sufficiently stable and yet sensitive enough to react completely at room temperature with hydriodic acid when the two are present in equivalent amounts. These requirements appear to be met by potassium permanganate. A potentiometer is employed, consisting of a rheostat having a coil 425 mm. long, made of about 650 turns of oxidised resistance wire closely contact-wound, and with a resistance of 169.45 ohms. A section of square brass beam, accurately graduated in mm., carries the sliding contact, and the contact points are narrowed so as to touch only two wires at once. Since the voltage of the titration cell never exceeds about 0.8 in

these measurements, the calculated resistance, 229 ohms, was added by means of a resistance box to the end of the instrument not traversed by the sliding contact. This makes the scale virtually 1 m. in length, and the value of 1 mm. equals approximately 2 millivolts when one storage cell is used as a balance. Calibration of the potentiometer without the extension to every 5 cm., together with the results of measurements of known voltages, showed that the error was nowhere greater than 0.4 per cent., while in the region of measurement it would be smaller, and with its virtual 1,500 turns of wire still smaller. A damped movable coil galvanometer, with rated sensitiveness 106 megohms, was used and was not found too sensitive; with this were used the Leeds and Northrup electric lamp and etched scale. The titration vessel consisted of a three-necked Woulff's bottle with a hole bored for the tip of the burette; one neck carried the drawn-out tube for the washed carbon dioxide or compressed air for vigorously stirring the solution, and the others the usual calomel electrode with a normal potassium chloride solution and the small, bright, platinum electrode. By means of this apparatus it is possible to titrate dilute sulphuric acid solution of iodine accurately with standard permanganate solution; chloride or bromide retards and diminishes in amount the sudden rise in voltage to an extent proportional to its concentration, but the presence of chloride in amount at least equivalent to the iodide, or one-fourth of this quantity of bromide, is permissible. Dichromate and iodate in 0.02 *N* and 0.05 *N* solutions may be titrated accurately by adding the solution to an excess of iodide in sulphuric acid and titrating the excess with permanganate, dichromate showing its theoretical oxidising capacity; silver has been determined in the same way. The high results obtained by Crotogino (*Zeitsch. anorg. Chem.*, 1899, **24**, 225) in titrating iodide with permanganate are not explainable by formation of iodic acid before the end-point was reached.

T. H. P.

Use of Spot Reactions in Qualitative Analysis. F. Feigl and R. Stern. (*Zeitsch. anal. Chem.*, 1921, **60**, 1-43.)—Tests made by adding a drop of the solution under examination to a drop of reagent placed previously on a plate, or, preferably, on a piece of filter-paper, are of wide application, and are often more sensitive than similar tests carried out in test-tubes. The authors give numerous examples of the usefulness of the method in the detection of individual substances and certain constituents in mixtures. Reactions of aluminium, uranium, and chromium are described, depending on the fact that salts of these metals yield coloured lakes with alizarin; manganese hydroxide yields a blue coloration with benzidine acetate solution.

W. P. S.

Separation of the Phosphorus from the other Components of Steel. R. Ariano. (*Gazzetta*, 1921, **51**, I., 1-31.)—The methods employed to separate phosphorus from the other constituents of steel are examined, the reactions involved being considered with the help of chemical statics and dynamics, and the conditions necessary to render this separation complete indicated. Following a discussion of the various methods employed for the elimination of the silicon and of the copper, arsenic, etc., the acetate method and the different modifications of the phospho-

molybdic method for precipitating the phosphorus are examined. In separating the silica, 16 c.c. of nitric acid of density 1.2 are used for the treatment of each grm. of steel, and, after each evaporation to dryness, the residue is taken up in hydrochloric acid of medium concentration. Ammonium phosphomolybdate does not appear to be a definite compound, and the ratio of the amount of P_2O_5 calculated from the weight of precipitate found on the basis of Carnot's formula, $P_2O_5, 24MoO_3, 3(NH_4)_2O, 3H_2O$, to the amount actually present, depends on the concentration of the molybdic anhydride, the quantity of phosphorus present, the temperature at which the precipitation takes place, the duration of the precipitation, stirring during precipitation, the nature of the metals present, the volume of the liquid, the nature and amount of the acids present, the concentration of the ferric ions, and the proportion of ammonium nitrate in the solution (see Jørgensen, *Zeitsch. anal. Chem.*, 1907, 370). The conditions favouring the precipitation are as follows: (1) High concentration of the molybdic anhydride; this, however, renders the precipitate less pure, so that the concentration must be regulated according to the subsequent treatment of the precipitate. (2) High temperature, even 100° C. being advisable provided that the arsenic has been previously removed; precipitation for twenty-four hours at the ordinary temperature does not yield very accurate results. (3) Stirring of the liquid during precipitation. (4) The solutions should be as dilute as possible, especially as regards iron. (5) The solution should be either neutral or very strongly acid, medium acidity rendering the precipitation incomplete. (6) The precipitate should be washed, not with nitric acid, but with water. (7) The same conditions should always be employed. (8) Addition of ammonium chloride, sulphate, etc., facilitates the precipitation and renders it more complete. T. H. P.

Volumetric Estimation of Arsenious Compounds by Means of Potassium Dichromate. R. Meurice. (*Ann. Chim. anal.*, 1921, 3, 85-86.)—The arsenious compound in hydrochloric acid solution is treated with potassium bromide, and the mixture then titrated with standardised potassium dichromate solution. During the titration a current of air is aspirated through the mixture, and then conducted into a test-tube containing potassium iodide solution and starch; as soon as all the arsenious acid has been oxidised, the next drop of dichromate solution added liberates bromine, which is carried over with the air into the iodide-starch mixture, in which it produces a blue coloration. W. P. S.

Separation of Tin and Antimony in Hydrochloric Acid Solution by Means of Hydrogen Sulphide. G. Luff. (*Chem. Zeit.*, 1921, 45, 249-251, 254-255, 274.)—With increasing amounts of concentrated hydrochloric acid, the temperature at which precipitation of antimony and tin salts by the treatment of their boiling solutions with hydrogen sulphide takes place, is lowered. Thus, with 8, 14, and 30 c.c. respectively added to 100 c.c. of solution, the temperatures are 102° , 102° , and 95° C. for antimony trisulphide; 102° , 100° , and 80° C. for the pentasulphide; and 90° to 95° , 75° to 80° , and 25° C. for stannic sulphide. The depression of the temperature is still more noticeable with ammonium chloride: with 36 grms. per 100 c.c., the trisulphide and pentasulphide are precipitated at 78°

and 60° C. respectively, whilst stannic sulphide does not separate, except after standing, even at normal temperature. The effect of varying concentrations of hydrochloric acid, with a fixed amount (16.5 per cent.) of ammonium chloride, on the separation of antimony and tin sulphides, has been investigated. Fourteen c.c. of hydrochloric acid (sp. gr. 1.193) per 100 c.c. of solution effects the clearest separation; the lowest amount to ensure separation is 8 c.c. per 100 c.c., and the maximum 35 c.c. per 65 c.c. of solution. Vortmann-Metzel's method is satisfactory for separation of antimonious sulphides, as well as modifications of it with 8 and 14 c.c. of hydrochloric acid respectively. Sufficiently accurate results are also obtained by the Panajotow-Prim method; if the ammonium chloride content exceeds 16.5 grms., however, the solution must be cooled in ice, introduction of hydrogen sulphide arrested at 20° C., and the solution filtered at 5° C.

W. J. W.

Estimation of Tungsten. G. Fiorentino. (*Giorn. Chim. Ind. Appl.*, 1921, 3, 56-58.)—The volumetric estimation of tungstic acid requires (1) a solution containing 32.6893 grms. of crystallised lead acetate and about 2 c.c. of acetic acid per 2 litres, and (2) a solution containing 7.485 grms. of crystallised ammonium molybdate per litre. If the tungstic acid is in the form of dissolved alkali salt, the solution, which should not contain excess of alkaline salts, is treated with 8 c.c. of concentrated ammonia solution; hydrated tungstic acid, however, is heated gently with 25 c.c. of water and 8 to 10 c.c. of concentrated ammonia until solution is complete. The solution in either case is diluted to about 200 c.c., acidified with acetic acid, then made alkaline to the extent of 1 or 2 drops of ammonia solution, and boiled until a litmus paper in the liquid turns wine-red. A measured volume, in excess, of the standard lead acetate solution is added to the boiling liquid, with continual stirring, boiling being continued until the precipitate becomes pulverulent or crystalline. Solution (2) is then added from a burette to the boiling liquid until a drop of the latter gives a yellow coloration with a drop of a fresh solution of 0.1 gm. of tannin in 10 c.c. of water. The volume (usually 0.3 c.c.) of the molybdate solution required to produce a distinct yellow coloration with the tannin is determined by a blank test on 200 c.c. of water and applied as a correction. The amount of WO_3 present is calculated by multiplying the amount of lead precipitated by the tungstic acid by 1.202. The method is based on the reactions: $\text{H}_2\text{WO}_4 + \text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2, 3\text{H}_2\text{O} = \text{PbWO}_4 + 2\text{C}_2\text{H}_4\text{O}_2 + 3\text{H}_2\text{O}$, and $\text{Mo}_7\text{O}_{24}(\text{NH}_4)_6, 4\text{H}_2\text{O} + 7\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2, 3\text{H}_2\text{O} = 7\text{PbMoO}_4 + 6\text{NH}_4\cdot\text{C}_2\text{H}_3\text{O}_2 + 8\text{C}_2\text{H}_4\text{O}_2 + 3\text{H}_2\text{O}$.

In the case of an ore or concentrate, 1.5 gm. of the finely powdered material is heated to 60° C., and occasionally stirred for an hour with 100 c.c. of concentrated hydrochloric acid in a covered beaker. The beaker is then heated more strongly on a sand-bath or asbestos card, the liquid being stirred continuously for thirty minutes, and then occasionally until reduced to about one-half of its original volume, when it is treated with 20 c.c. of nitric acid and evaporated to 25 c.c.; 5 c.c. of nitric acid are then added, and the solution evaporated to 15 to 20 c.c., mixed with about 180 c.c. of boiling water, left for some hours, and then filtered, the precipitate being washed by decantation with 1 per cent. nitric acid. Unless a very exact result is required,

the filter and its contents are returned to the beaker and heated gently with 8 to 10 c.c. of concentrated ammonia solution and 25 c.c. of water, which should dissolve all the tungstic acid and leave no undecomposed particles of ore. Either all or an aliquot part of this solution, containing about 0.25 grm. of WO_3 , is treated according to the volumetric method described above. When great accuracy is necessary, the filter is washed with the mixture of 8 to 10 c.c. of ammonia and 25 c.c. of water into the beaker containing the bulk of the precipitate, the liquid being heated gently until all the tungsten trioxide is dissolved, filtered through the same filter, and the latter washed with very dilute ammonia solution. The liquid is then dealt with as above, and the residue on the filter dried and calcined at a moderate temperature, any tungsten trioxide it contains being estimated by the cinchonine method (see below). The acid liquor from the original attack of the ore and from the washing should also be concentrated and precipitated with cinchonine, since part of the WO_3 may be hydrolysed and dissolved if too little acid remains when the boiling water is added.

In the cinchonine gravimetric method, 0.5 to 1 grm. of the tungsten ore is heated to dull redness with a little wood charcoal, and about 1 grm. of sodium hydroxide, previously fused in an iron crucible. The fused mass is cooled and dissolved by boiling with water, finally with addition of 4 to 5 grms. of solid ammonium carbonate; the liquid is then filtered, the filter washed with very dilute sodium hydroxide solution, and the filtrate heated, treated gradually with 30 c.c. of concentrated hydrochloric acid, boiled for a few minutes, mixed with 8 to 10 c.c. of a solution of 30 grms. of cinchonine in 50 c.c. of concentrated hydrochloric acid and 250 c.c. of water, and allowed to stand for some hours. It is then passed through a filter containing a little pulped filter-paper, the precipitate being washed three or four times with dilute cinchonine solution (30 c.c. of the concentrated solution, 30 c.c. of concentrated hydrochloric acid, and 1,000 c.c. of water), and once only with cold water; the filtrate contains the tin, which may be estimated by the Pearce-Low method. The filter is introduced into the original precipitating beaker and heated gently, and stirred to break lumps, with 8 to 10 c.c. of ammonia solution and 25 c.c. of water, the solution being filtered and the filter washed with hot water containing a little ammonia. The filtrate is heated to expel the excess of ammonia, treated at once with 2 to 3 drops of hydrochloric acid and 8 to 10 c.c. of the concentrated cinchonine solution, and stirred well for some minutes. After one to two hours, the liquid is filtered through an ashless filter containing a little pulped filter-paper, and the precipitate washed as before with dilute cinchonine solution and once with water, dried, ignited in a platinum crucible and weighed as WO_3 . A small proportion of silica sometimes occurs in the precipitate, and this may be expelled by treating the precipitate with 1 to 2 drops of dilute sulphuric acid and a little hydrofluoric acid, the latter being then driven off, 1 to 2 drops of nitric acid added, and this and the sulphuric acid then expelled by heating. Cinchonine may be recovered from liquors containing it by rendering them alkaline with ammonia.

T. H. P.

New Method for Separating Ferric, Chromium, and Aluminium Hydroxides. M. and M. Lemarchands. (*Ann. Chim. anal.*, 1921, 3, 86-87.)—The three

hydroxides are precipitated together in the usual way, and washed until free from ammonia; the precipitate is then boiled with 10 per cent. sodium hydroxide solution, and sodium perborate is added. Ferric hydroxide remains insoluble and is separated by filtration, whilst the chromium and aluminium hydroxides pass into solution as sodium chromate and sodium aluminate respectively. The filtrate containing these two compounds is divided into two parts: in one, the aluminium is precipitated as hydroxide by boiling with the addition of an excess of ammonium chloride, and in the other portion the chromium is precipitated as lead chromate. W. P. S.

Iodimetric Method for the Estimation of Chromium in Chromite. E. Little and J. Costa. (*J. Ind. Eng. Chem.*, 1921, 13, 228-230.)—The substance is fused with sodium peroxide, and the resulting chromate estimated iodimetrically in hydrochloric acid solution, ammonium fluoride being added to prevent interference by ferric chloride. The details of the method are as follows: 0.4 gm. of the sample is mixed in an iron crucible with 3 grms. of sodium peroxide, the mixture is covered with a further 2 grms. of peroxide, then heated at a low red heat for five minutes, and fused for fifteen minutes at a higher temperature. After cooling, the fused mass is dissolved in 150 c.c. of water, the solution treated with a further 0.5 gm. of peroxide, and boiled to decompose the excess of the latter. The solution is then cooled, hydrochloric acid is added until the ferric hydroxide has dissolved, and 5 c.c. of concentrated acid are introduced for every 100 c.c. of solution. Ammonium fluoride is now added until the solution no longer gives a reaction for ferric iron with potassium ferrocyanide, an excess of 1 gm. of ammonium fluoride is introduced and, after the addition of 3 grms. of potassium iodide, the liberated iodine is titrated with standardised thiosulphate solution. W. P. S.

Entanglement of Lime and Magnesia by Chromic Oxide Precipitates. E. Toporescu. (*Comptes rend.*, 1921, 172, 600-602.)—The proportion of lime contained in the chromic oxide precipitated by ammonia solution from a boiling solution containing calcium and chromic chlorides tends towards a limit corresponding with formation of calcium chromite, $\text{Cr}_2\text{O}_3, 3\text{CaO}$; the lime may be removed by washing the precipitate on the filter with boiling 5 per cent. ammonium nitrate solution. Similarly, the proportion of magnesia carried down with chromic oxide from a solution containing magnesium and chromic sulphates and ammonium chloride is variable and, if the liquid be saturated with the ammonium salt, reaches a limit corresponding with the formula $\text{Cr}_2\text{O}_3, 3\text{MgO}$; the magnesia is removable by washing the precipitate several times by decantation with boiling 5 per cent. ammonium nitrate solution. T. H. P.

The Quantitative Estimation of Calcium and Magnesium in Various Salt Mixtures. E. Canals. (*Bull. Soc. Chim.*, 1921, 29, 152-158.)—The precipitation of calcium and magnesium as oxalates in ammoniacal and acetic acid solutions has been studied. The essential condition for precipitation of lime in the cold in presence of magnesium is that a very dilute solution (not more than 1 part in 400) be taken. Magnesium is not precipitated with calcium oxalate when the dilution of

magnesium is greater than 1 : 100, and magnesium oxalate can be removed from the precipitate by repeated washing with boiling water, in some cases as much as 500 c.c. being used. In acetic acid solution the lime can be accurately estimated if the concentration is about 1 : 700, and the precipitate be well washed with boiling water.

H. E. C.

Removal of Nitrates by Means of Alcohol. R. Schneidewind. (*Chem. and Met. Eng.*, 1921, **24**, 22; *J. Soc. Chem. Ind.*, 1921, **40**, 131A.)—Nitric acid and nitrates are removed by heating with alcohol and sulphuric acid. For example, 20 c.c. of nitric acid are diluted with 150 c.c. of water and treated with 15 c.c. of sulphuric acid, and when the liquid is nearly boiling 5 c.c. of ethyl alcohol are carefully run in from time to time until nitrous fumes cease to be evolved on adding more alcohol. The liquid is boiled until free from alcohol, and is then sufficiently free from nitrates not to react with ferrous sulphate or oxidise hydrogen sulphide.

R. G. P.

Estimation of Nitrous Acid in Mixed and Waste Acids. H. Toussaint. (*Z. angew. Chem.*, 1921, **34**, 102.)—A Woulff bottle with three necks is used for the estimation, through the outer ones of which pass a funnel, and an inlet tube for carbon dioxide. Seven hundred c.c. of air-free, distilled water is first placed in the bottle, and the air in this is displaced by carbon dioxide. The acid is then introduced through the centre neck from a burette, after which a glass tube, 10 cm. long, is fixed in the neck by means of a rubber sleeve, so that its lower end is level with the top of the Woulff bottle. Potassium iodide solution is run in through the funnel, and the mixture is then titrated with $\frac{N}{2}$ or $\frac{N}{10}$ thiosulphate, introduced through the centre neck, a stream of carbon dioxide being passed through during the operation, for which purpose the inlet tube should have been previously pushed down to the bottom of the bottle. Starch solution may be used as indicator if desired. Large amounts of ferric oxide in the acid affect the results; the iron must therefore first be removed by sodium hydroxide, free from nitrate, and the filtrate subsequently acidified with pure sulphuric acid.

W. J. W.

PHYSICAL METHODS, APPARATUS, ETC.

Electric Oven for Rapid Moisture Estimations. G. L. Spencer. (*J. Ind. Eng. Chem.*, 1921, **13**, 70-72.)—In an electric oven the material is enclosed in a crucible, preferably of metal, having a bottom of Monel metal filter cloth. The crucible is in close contact with its seat and above an annular channel connecting several crucible openings, and is in communication with a vacuum pump or ejector, by means of which electrically heated air is drawn through the oven. The heating element for the oven is placed inside to prevent loss by radiation; the temperature is regulated by means of a rheostat, and a time-switch rings a bell at the end of the drying period. The apparatus is suitable for drying any substance through which air can pass freely, and is especially adaptable to the drying of raw sugar and cane bagasse; with these latter materials tests were completed in ten and thirty minutes respectively.

W. J. W.

Laboratory Apparatus. G. Ajon. (*Giorn. Chim. Ind. Appl.*, 1921, **3**, 62-63.)—*Extractor with Continuous Flow of Liquid.*—This consists of a modification of the Soxhlet extractor, in which the syphon tube, just after it begins its downward course, is inserted into a wider tube connected in the ordinary way with the stem of the apparatus. *Multiple Hot Filter.*—This consists of an elongated, constant-level, copper water-bath, which has a number of small holes in the base and corresponding larger holes in the lid to take funnels, and is connected by means of two tubes with the upper and lower parts of a small boiler, so that continuous circulation of water takes place. It is intended especially for the filtration of calcium citrate in analyses by Warington's method. T. H. P.

Cold Test Apparatus for Oils. G. H. P. Lichthardt. (*J. Ind. Eng. Chem.*, 1921, **13**, 145-146.)—The apparatus consists of an inclined, galvanised iron refrigerator tank, 6 × 6 × 6 inches, in which are placed glass tubes of 0.3 inch internal diameter, which project appreciably through one side of the tank, and are bent upwards just beyond the other side and connected with an air supply. The samples of oil are put in the tubes so as to occupy lengths of about 6 inches, and the tank is then filled with a freezing mixture of acetone and carbon dioxide snow. The temperature at which, under an air-pressure of 16 inches of water, the oil becomes solid and fails to move is noted, after which the temperature is allowed to rise slowly, readings being taken at short, fixed intervals. The temperature at which the oil appears in the projecting portions of the tubes is then taken as the "cold test." W. J. W.

Melting-Point Apparatus. F. Friedrichs. (*Zeitsch. angew. Chem.*, 1921, **34**, 61.)—An apparatus for melting-point determinations consists of an outer glass vessel containing the heating liquid, and an inner one surrounding the thermometer, which serves to eliminate the error due to lag. Both vessels are oval in shape, but have flattened front and back surfaces, which enable microscopical readings to be made without interference due to curvature of the glass. Tubular extensions, fused obliquely into the base of the outer vessel, admit of the melting-point tubes being placed in position through openings in the inner vessel without removal of the thermometer. W. J. W.

Determination of Boiling-Point of Minute Quantities of Substances. V. Arreguine. (*Ann. Chim. analyt.*, 1921, **3**, 40-49.)—The method, which is similar in principle to that of Schleiermacher, is based on the vapour tension of the substance, and can be employed in cases where only a few mgrms. of material are available. The apparatus consists of a test-tube about 80 to 85 mm. in length and 9 mm. in diameter, which is half-filled with mercury, and a piece of open tubing (about 5 mm. in diameter) is selected of such bore that when immersed in the mercury the meniscus in the open tube is level with that in the test-tube, thus eliminating error due to capillarity. One end of the open tube is then sealed and blown into a small bulb, so that it fits inside the test-tube, leaving an annular space of 0.5 to 1 mm., and the tube is cut to a length of about 52 mm. For the determination of boiling-points, the inner tube is filled with mercury to within about 1 mm. of the top; a drop of

the liquid is then added, precautions being taken (*e.g.*, gentle heating) to eliminate air from the tube and its contents. In the case of solids, a small quantity of the substance is melted in the bulb and solidified by cooling before filling the tube with mercury. The filled tube is inverted and placed inside the test-tube, the open end being submerged below a short column of mercury in the test-tube. The inner tube is then fixed in position by filling the space in the test-tube above it with glass beads, and covering with a metal cap clipped over the rim of the test-tube. The test-tube, with its inner tube, is then fixed near a thermometer in a bath, which is heated as evenly as possible; on raising the temperature, the point at which the meniscus of the mercury in the inner tube containing the substance is level with that in the test-tube is taken as the boiling-point. The chief difficulty of operation is that due to local inequalities in temperature of the heating bath. The method only yields approximate results, and is unsuitable for substances boiling above 210° to 220° C., owing to errors produced by the vapour pressure of the mercury; fusible alloys were found to be unsuitable as substitutes for mercury. Formulæ are quoted for the correction of errors due to the vapour tension of the mercury and to excess of liquid above the mercury in the inner tube. The following figures were obtained without corrections: Water, 99.6° ; carbon disulphide, 46° ; ethyl alcohol, 78.1° ; ether, 34.96° ; aniline, 180.3° , 183.5° ; acetone, 55.8° ; and monobrombenzene, 153° C. The calculated corrections for a column of 1 mm. of liquid above the mercury in tube were generally very small (0.001° to 0.005° C.) and therefore negligible. The following corrections were calculated for the vapour tension of mercury: With water, 0.015° ; with carbon disulphide, 0.001° ; with alcohol, 0.005° ; with monobrombenzene, 0.17° ; with aniline, 0.64° C.

R. G. P.

Determination of the Flash Points of Mixtures of Air and Benzene.

W. Reinders. (*Chem. Weekblad*, 1921, 18, 157-159.)—An apparatus for determining the maximum and minimum flash points of air saturated with benzene vapour consists of a glass tube of 35 cm. length and 2 cm. diameter, having two platinum wires fused into the top part, and terminating at its upper end in a narrow tube with funnel and tap. It is surrounded for the greater part of its length by a glass water-jacket, provided with stirrer and thermometer, and is connected by rubber tubing with a levelling tube. The tube is first filled with water, after which, by manipulation of the levelling tube, a layer of about 10 cm. of benzene and a few cubic centimetres of air are introduced. The water in the jacket is brought to any desired temperature, and the apparatus is allowed to stand for five minutes to ensure saturation of the air with benzene vapour. An electric spark is then passed through the mixture. For determinations at temperatures below 0° C., the jacket is filled with a freezing mixture, and the benzene layer must fill the whole portion of the tube which is enclosed in the jacket, or alternatively, the water below the benzene layer must be superseded by a saturated salt solution. Results obtained with various benzene fractions are tabulated. The boiling-points of the fractions themselves may vary between wide limits, and consequently afford but little information as to the volatility of the benzene; a specific gravity determination must therefore be made.

W. J. W.

Experimental Data and Balance for Fixing the Exposure to X-Rays in Radiography and Radiotherapy. F. Miramond de Laroquette and S. Millot. (*Comptes rend.*, 1921, 172, 525-527.)—The results obtained by the authors do not confirm the proportionality of the chemical or biological effect of X-rays to the square of the voltage, or to the square of the spark-length. Tests made with various forms of tubes and apparatus show, indeed, that for purposes of calculation it is preferable to retain the degree element B , and to measure this directly with a radiochromometer, and not by the spark-length, the variations in the two cases being sometimes discordant. When the radiation increases by 1 B unit, the effect produced superficially and in depth increases on the average by about 33 per cent. between 4 B and 7 B . The absorption of X-rays by different soft tissues, such as skin, fat, and muscle, is virtually the same, aluminium exhibiting a tenfold greater coefficient of absorption; theoretically, the aluminium used for filtration in radiotherapy should not be more than 3 mm. in thickness. For the various factors involved in the calculations, graphic scales have been constructed, these being arranged on a plate oscillating about a horizontal axis. Equal weights are placed on the scales at the graduations corresponding with the data fitting the particular case, a similar weight being then moved along the scale of the unknown factor until equilibrium is established. The different formulæ, being calculable by means of logarithms, may thus be transformed into equilibrium equations: By this means it is possible to determine instantly the necessary exposure for any radiographic or radiotherapeutic case, to make any combination by varying the current and voltage, the distance of the tube and the thickness of the filter, and by interposing a screen, and also to ascertain in Holzknicht units (H) the quantity of the incident radiation and also that which is absorbed and thus acts at any particular depth in the tissues. T. H. P.



REVIEWS.

L'ÉQUILIBRE DES SUBSTANCES HÉTÉROGÈNES. Abridged account by WILLARD GIBBS, translated by G. MATISSE, with complete explanatory notes. Pp. 102. Paris: Gauthier, Villars et Cie. 1919. Price 3 fr. 50, plus 50 per cent.

The well-known treatise of Gibbs on heterogeneous equilibrium has already been translated into French, but it is difficult for the non-mathematical reader to understand the abstract manner in which the subject is expounded. In 1878, in the *Scientific American*, Gibbs published an abbreviated account of the essentials of his theory of heterogeneous equilibrium and set out the principal formulæ. The present volume is an exact translation of Gibbs's synopsis, together with notes by the translator explaining the derivation and application of the formulæ given in the text.

The book is addressed to those who have not the mathematical equipment necessary to understand the larger treatise and to those who, having mastered the large work, wish to summarise and retain the principal propositions. The notes are very full and clear, and should enable the reader, who is possessed of the necessary

quantum of mathematics which is essential for the understanding of modern physical chemistry, readily to grasp the fundamental theory of heterogeneous equilibrium.

H. E. Cox.

PUBLIC HEALTH CHEMICAL ANALYSIS. By ROBERT C. FREDERICK and AQUILA FORSTER. Pp. 284 and Appendix. London: Constable and Co. Price 21s.

The subject-matter of this excellently printed volume includes the analysis of air, water, sewage, and trade effluents, most of the ordinary foods, condiments, and drinks, disinfectants, soaps, and rag flock, and as the whole ground is covered within the compass of 286 pages, it will be obvious that the authors have had to exercise both discrimination and restraint. Only in relatively few cases could alternative methods be included, and much discussion of theoretical principles could not be expected.

In spite of these limitations the authors have acquitted themselves well, and have produced a volume which is essentially practical and extremely clearly written.

Judging from its title the book is intended mainly for those preparing for the Diploma of Public Health, and if this be so it will account for the relatively large amount of space devoted to the analyses of air and water, which between them occupy nearly one-third of the total number of pages. It may, perhaps, also account for the insertion of an introductory chapter, describing the fundamental operations of analysis, weighing and measuring, the care of platinum dishes, the use and preparation of normal solutions, etc. Except for the revolutionary recommendation that "small quantities of infusible precipitates, such as barium sulphate, should be weighed direct on the pan of the balance," no exception can be taken to the instructions given, but surely they should be quite unnecessary for those competent to undertake the wide range of analytical operations described.

In dealing with the examination of air, the writers seem to have been carried away by their partiality for the Haldane apparatus, to which some twenty pages are allotted, and undue space is given to the relatively simple determination of carbonic acid. Haldane's hæmoglobin method for the determination of carbon monoxide is a dangerous one to put into the hands of any but experts. It sounds delightfully simple, but only the happy possessors of a retina extremely sensitive to the red end of the spectrum can hope to apply it with any chance of success. The authors warn the reader that experience is required, but unless the eyes of the operator possess the requisite red sensitiveness no amount of experience will suffice.

The chapter on water is commendably full, and a series of fourteen typical analyses of waters from various sources, with appropriate criticisms of the results, will be of considerable help to the beginner.

An alternative to the phenol-sulphonic acid method for the determination of nitrates might with advantage have been given, and the recommendation to evaporate 5,000 c.c. of the water to dryness for the estimation of the silica and the ordinary bases is obviously a counsel of perfection which it would be very inconvenient to follow.

The section on milk and milk products is well up to date, but with regard to matters of detail the writers here again show a tendency towards the academic, and the

procedure recommended is often unnecessarily laborious and expensive. The determination of total solids in milk by weighing 10 c.c. into large platinum dishes, each containing $3\frac{1}{2}$ inches of platinum wire (to break up the skin that forms on heating) would hardly commend itself to the busy practitioner in these hard times. The omission of such an accurate, simple, and widely applicable method as that of Gottlieb for the determination of fat is to be regretted even though the Adams, Werner-Schmidt maceration and two centrifugal processes are described.

Another rather more serious omission is the failure to mention the use of powdered pumice in the Reichert-Polenske process, as the employment of broken porcelain, which the authors recommend, might seriously vitiate the results.

To the other articles of food, etc., less space is allotted, but there is a chapter on the general methods of examination of sugars, which on the whole is very thorough and systematic, and the treatment of the problem of the analysis of mixtures of various sugars is much in advance of what is found in much more pretentious works. There are, however, one or two statements of a misleading or otherwise objectionable character to which attention should be drawn. The Clerget formula (for an angular degree instrument) is given as $S = \frac{100 D}{95 - \frac{D}{5}}$ where D is stated to be the "angular deviation" of 1 dem. of the solution after inversion. The expression of the algebraical sum of the readings before and after inversion by the single symbol D, coupled with the ambiguous term "angular deviation," might easily mislead the unwary, although the matter is more clearly stated on a later page. The verbal epicure would also be shocked by the use of the term "negative rotation" to signify lævo-rotation, and of the objectionable word "opticity" for rotatory power.

The statement that in the manufacture of beer the malt is digested with water for two or three hours at 30° C. is not in accordance with facts, as the temperature usually ranges between 61° and 68° C.

In the case of tea and pepper the student is told to determine the fibre by acid treatment alone; under Cocoa the fibre is not mentioned at all.

In dealing with the Marsh-Berzelius process no precautions are recommended to secure the sensitiveness of the zinc. It is to be regretted that no reference is made to the much simpler and now official Gutzeit process.

The illustrations are excellent and to the point, but the photomicrographs of starches, condiments, etc., suffer from an unnecessarily high magnification, and, as is generally the case, hardly justify their existence and are quite unnecessary if, as should be the case, authentic specimens of the substances are examined.

There are very few misprints, the arrangement and general style of the volume is excellent, and taken as a whole the work is a credit to authors and publisher alike.

C. H. CRIBB.

DAIRY CHEMISTRY: A PRACTICAL HANDBOOK FOR DAIRY CHEMISTS AND OTHERS HAVING CONTROL OF DAIRIES. By HENRY DROOP RICHMOND, F.I.C. Third Edition, Revised and Reset. London: Charles Griffin and Co., Ltd. 1920. Price 25s.

The last edition of this standard work was published in 1915, and the appearance of a new edition in such a comparatively short period as five years indicates

the value of the book. The general plan of the book remains the same, but advantage has been taken of the present revision to collect scattered items into the part allocated to the particular subject and to rearrange other details in a more useful order.

The book is now divided into three parts dealing respectively with the Constituents of Milk, the Analysis of Milk and Milk Products, and Technical Applications. Each of these parts is full of information, the latest researches have been included, and new work appearing while the book was in the press is given in addenda.

An improvement, which adds greatly to convenience in using the book, is the elimination of the cumbersome folding tables, these being now incorporated in the text.

One or two of the illustrations are poor, particularly those showing the lactometer (p. 75) and butter and margarine under polarised light (p. 270), and those who are desirous of seeking further information on any point will possibly regret that all references to literature are omitted from the text.

Mr. Richmond is to be congratulated in that he has improved a book which had already a world-wide reputation as a standard. In addition to dairy chemists, food analysts in general will find the book to be a valuable addition to their libraries.

W. P. SKERTCHLY.

RAPID METHODS FOR THE CHEMICAL ANALYSIS OF SPECIAL STEELS, STEEL-MAKING ALLOYS, THEIR ORES, AND GRAPHITES. By C. M. JOHNSON. Third Edition.

Pp. xi + 552. New York: John Wiley and Sons; London: Chapman and Hall. 1920. Price 36s. net.

The first edition, numbering 221 pages, was reviewed by L. Archbutt in the *ANALYST* of 1909, p. 252; in the present work the increase of the text matter over the second edition represents more than 100 pages. The processes described for testing special steels and steel-making alloys are the result of the practical experience of a steel works chemist; as such they should prove a valuable guide to other analysts working in this field, and a careful perusal of the book will disclose a number of helpful suggestions. The same cannot always be said of the processes recommended for analysing ores of the elements which enter into the composition of special steels. In this class of work many of the methods used by the author are laboursome, involved, and anything but "rapid." To take one example, that of wolframite: the decomposition by strong hydrochloric acid of the ore "ground to the finest flour" requires far less than six hours, whilst the use of a double filter and addition of "a three-quarter inch ball of filter pulp" to the tungstic acid "to hasten filtration and secure a perfect washing" are not only unnecessary, but even detrimental in this case. The excess of filter pulp containing the tungstic acid from 1 gm. of ore must be given "not less than sixty washings" to remove the potassium salts derived from the chlorate, which is used as an oxidiser. Nitric acid would answer just as well without introducing any fixed constituents.

The author next commits what the reviewer regards as an error of judgment in igniting the impure precipitate and weighing it "as WO_3 plus some Fe_2O_3 , Al_2O_3 , SnO_2 , Mn_2O_4 , CaO , Ta_2O_5 , Nb_2O_5 , MoO_3 , CuO , and all of the SiO_2 ," and then

evaporating with hydrofluoric acid to eliminate silica even if it reaches "from 30 to 60 per cent., as it often does in unconcentrated ores and slimes"; incidentally, the *aqua regia* method does not give good results with low-grade ores. Considering that the bulk of such silica is usually present in the form of quartz, which is not easily attacked and volatilised, it would be far wiser to eliminate it before ignition—*e.g.*, by dissolving the tungstic acid in ammonia. The tungstic acid is then weighed, fused, and submitted to a series of manipulations the object of which is to extract and weigh the impurities, tungstic acid being found by difference. Removal of the impurities followed by direct weighing of the purified tungstic acid appeals to the writer as being more rapid and reliable. For the quick and accurate estimation of tin in tungsten ores, the author would be well advised to refer to and adopt Powell's excellent method (*J. Soc. Chem. Ind.*, 1918, **37**, 285*r*). Arsenic is determined in tungsten ores by weighing as pentasulphide; a volumetric method would be preferable for speed and accuracy.

On p. 197 iron is titrated in presence of titanium after reduction with zinc, though on p. 52 this is rightly stated to be inadmissible. In the analysis of crude zirconia the precipitate obtained by sodium phosphate and washed "at least sixty times with water" is described as "pure phosphate of zirconium." This can hardly be the case unless zirconium is precipitated by ammonium phosphate from a solution free from alkalis, and the precipitate washed with ammonium nitrate solution; on ignition it yields pyrophosphate, ZrO_2 , factor 0.4632 (Lundell and Knowles, *J. Amer. Chem. Soc.*, 1919, **41**, 1801). A general criticism must be directed against the author's habit of washing a precipitate forty, sixty, or even eighty times.

The book is characterised by lack of systematic arrangement and a wealth of minute instructions: the more or less detailed description of the precipitation of molybdenum sulphide occurs more than ten times. In every case the sulphide is roasted to trioxide, which is weighed, whilst the more reliable estimation as lead molybdate is described once as a check method.

Misprints and errors in the text are not infrequent; the author's style is often careless and distinctly Transatlantic—*e.g.*, "the crucible is attacked some by the niter" (p. 327). The following quaint direction is worth remembering in case of a sudden shortage of litmus paper: "Wash . . . until the outside of the filter has no sour taste, especially along the double thickness" (p. 319).

The index is rather poor, the references under each letter not being arranged alphabetically.

In short, the book contains much that is of value, but in a crude state. It would be greatly improved by extensive excision and careful revision.

W. R. SCHOELLER.

RECOVERY OF NITRATE FROM CHILEAN CALICHE. By A. W. ALLEN. Pp. xvi + 50. London: Charles Griffin and Co., Ltd. Price 6s. net.

In a preface the author points out that, although the Shanks system of caliche treatment has effected notable economies in the Chilean nitrate industry, its inefficiency is manifesting itself more and more as the richer deposits become exhausted, since it cannot be successfully applied for the recovery of nitrate from low grade caliche

and waste products (ripio). Estimates of the latter in numerous dumps vary between 100,000,000 and 150,000,000 tons, whilst the quantity of the former probably exceeds a billion tons. The fact is emphasised that only by successful and economical treatment of these at present valueless products can the nitrate industry be placed on a secure footing and enabled to compete with the steadily progressing synthetic processes for production of nitrogen compounds; nevertheless, the present position of the latter industry tends to negative the author's implied denial on p. 19 of its commercial feasibility. In the first part of this volume the Shanks process is described; its defects are said to be: high loss of nitrate in ripio and borra, high cost of operation and heavy overhead charges, excessive labour requirements, and inaccurate accounting of yields. The latter part of the book deals with the new Allen extraction process, which is briefly described. Its advantages are: even percolation on account of homogeneity of the charge, improved and shorter treatment, better displacement and more rapid solution, possibility of recovery of nitrate from waste products, and an increased yield up to 90 to 95 per cent. Alternative methods for evaporation and crystallisation are also discussed. The information is clearly conveyed and a glossary of Spanish terms forms a useful guide, but the index might, with advantage, be reconstructed. The book should serve to call fresh attention to the imperative necessity for improved methods in treating caliche, whilst, at the same time, the lines along which development should proceed are indicated.

W. J. WRIGHT.

A DICTIONARY OF APPLIED CHEMISTRY. Volume I.: A to Calcium, by SIR EDWARD THORPE, C.B., F.R.S., assisted by eminent contributors. New and revised edition, 1921. London: Longmans, Green and Co. Price 60s. net.

It is clearly impossible for a reviewer to comment adequately on all the sections of such a comprehensive and authoritative treatise as Thorpe's "Dictionary of Applied Chemistry." The names of the various writers and the established reputation of the book are sufficient guarantee of its general excellence.

The new edition is a distinct improvement on the last (1912) edition, and contains so much new matter that it is proposed to issue six or seven volumes instead of five; the scale of the revision and enlargement may be judged by the expansion of A to Calcium from 614 to 752 pages. Several articles have disappeared or been transferred to other headings in later volumes and a number of new ones included; the whole is brought up to date so far as is possible in so large a work. References to literature are up to 1919, and atomic weights are those of 1920. Paper and binding are of similar good quality to the 1912 edition.

The article on Acetic Acid and Vinegar manufacture has been rewritten by C. A. Mitchell, and indicates a notable advance in this industry. A few obsolete passages, however, have escaped excision; thus on page 21 it is remarked that perry and crab-apple vinegar are used in Wales and Monmouthshire; no evidence of this has been observed by the writers in recent years.

Under Acetone details of the new catalytic processes are not given for reasons, stated in the Preface, of inexpediency.

Some of the articles are not so up to date as others. The section on Acidimetry appears to the reviewers to be lacking in this respect; references are only to 1915 and the portion dealing with indicators does not give their $[H^+]$ ranges and exponents. A table of the $[H^+]$ values at which the colour changes would be most useful, much more so than the rather old-fashioned table on page 59. The bibliography under Indicators should certainly include Prideaux's excellent book on the theory and use of indicators; similarly the general references (p. 64) should be more recent and include the 1911 edition of "Sutton."

Under Alkaloids a new article appears by Barger incorporating part of the article on Vegeto-Alkaloids of the 1912 edition with a general account of this group of substances. The article on Aluminium has been extended, but it is singular that no mention is made of the phosphate method of estimation, although the hydroxide precipitation is described. On page 171 there occurs a reproduction of the 1912 edition of the misspelling of Le Chatelier's name.

The section on Analysis, like that on Acidimetry, is not sufficiently revised; references are to old editions of well-known textbooks. Mellor's textbook of quantitative analysis is not included in the bibliography. Under Assaying three figures of commonplace implements have been deleted and more valuable information inserted, but the section on the assaying of coal is so sketchy as to be of little value; the determination of calorific power by mixture with potassium chlorate and nitrate in Thompson's calorimeter is obsolete; this subject might well be left for adequate treatment in a later volume under Fuel.

The subject of radioactive constituents of the atmosphere has progressed considerably of recent years, so that the article containing only references to 1909 is not quite worthy of a 1921 edition.

There is a valuable and excellent fourteen-page addendum to the article on Balance, dealing with highly refined weighing and discussing all factors affecting this rather difficult matter; various types of micro-balance are clearly explained.

It may surprise many readers to find an account of the solution usually known as Fehling's described and discussed in detail under Barreswil's name. However accurate it may be to ascribe this solution to Barreswil, it is so long associated with Fehling's name that such change is rather startling.

The section on Boiler Incrustations, by Brame, is new and that on Brewing, by Stern, is much condensed, so that we now have a short and concise article which is highly desirable for a subject which for adequate treatment requires several volumes of its own. The numerous references to literature will enable a reader readily to obtain supplementary information on any desired point.

The work as a whole is beyond criticism, and the new edition deserves a place on the bookshelf of every technical chemist; there are remarkably few typographical errors, and great care has evidently been taken by the Editor. Of the many references turned up by the reviewers, taken at random, not one was found incorrect.

G. B. THOMPSON.

H. E. COX.

TECHNICAL CHEMISTS' POCKET BOOK. By ROBERT ENSOLL. London: E. and F. N. Spon, Ltd. Price 8s. 6d. net.

The one hundred and ninety pages of this book contain an immense amount of material useful to technical chemists, metallurgists, and chemical engineers, the aim of the author being to produce a complementary volume to the well-known "Bayley's Chemists Pocket-Book." The greater part of the information is given in tabular form and the scope of the work may be judged from the following selection of subjects taken from the contents: Weights, areas, etc., calibration of storage tanks, combustion data, furnace construction, fuel consumption, boilers, properties of metals, strength of materials, alloys, thermal and electrical conductivities, specific gravities of solutions, metals, solids, etc., compression of gases, fuse wires, injurious effects of various gases, and typical analyses.

The tables giving the strength of alcoholic solutions from the specific gravities do not show a greater alcoholic strength than 42.18 per cent. by weight, and would be of enhanced value if they were extended to include stronger alcoholic solutions.

References to literature are up to date and but few errors have been noticed in the text. The name "Inchley" at the bottom of page 63 should be "Hinchley"; on page 124 the expression "< 99" under the heading of tin should be "> 99."

The book will be found extremely useful for reference, whether it is carried in the pocket or used on the table or bench, but it is a pity that the paper is not of better quality, as this affects the clearness of the printing.

W. P. SKERTCHLY.



MINISTRY OF HEALTH.

REPORT OF THE DEPARTMENTAL COMMITTEE ON THE CONTROL OF CERTAIN THERAPEUTIC SUBSTANCES, 1921 (Cmd. 1156).

THE substances included in the terms of reference were classified into three groups: (A) Bodies described in the U.S. Regulations of 1919 as "biologic products,"—i.e., vaccines, sera, toxins, antitoxins, and analogous products. (B) Synthetic remedies, such as salvarsan and its analogues. (C) Substances corresponding more nearly with the popular definition of ordinary "drugs"—e.g., preparations of digitalis, strophanthus, squill, ergot, cannabis indica, pituitary gland, etc.

An account is given of the present position as regards standardisation and control of substances which cannot be adequately tested by direct chemical means, together with an outline of the systems of control adopted in U.S.A. and Germany.

Evidence was given before the Committee by scientific authorities and representatives of commercial and manufacturing interests, and the following is a summary of the principal recommendations based upon this evidence:

- (a) That therapeutic substances which cannot be tested adequately by chemical means should be subject to supervision and control.
- (b) That the Controlling Authority should be the Committee of the Privy Council which was appointed by an Order in Council dated 11th March, 1920, under powers conferred by the Ministry of Health Act, 1919.

- (c) That the Controlling Authority should be assisted by an Advisory Committee which might consist of members nominated by the Minister of Health, the Secretary for Scotland, the Chief Secretary for Ireland, the Naval and Military Authorities, the General Medical Council, the Medical Research Council, and the Pharmaceutical Society.
 - (d) That there should be a Central Laboratory under Government control, wherein standards would be prepared and maintained, research carried out in connection therewith, and tests made to ascertain that the products issued by manufacturers conform with the standards laid down. The Medical Research Council, who already possess the requisite organisation, should be responsible for this Central Laboratory.
 - (e) That the method of control should include the licensing of manufacturers, inspection of their plant, premises, and processes, and the testing of the finished products.
 - (f) That, with certain exceptions, testing by the Central Laboratory should be confined to samples taken from makers' stocks or bought in the open market, leaving to the manufacturers the primary responsibility for securing that the products conform with the prescribed standards and tests. That power should, however, be taken to require a manufacturer to submit for central testing, for a stated period, samples of every batch of a substance made.
 - (g) That power should be taken to inspect all premises where the substances are made and the several processes of their manufacture, but that in the case of salvarsan and its analogues (Group *B*) and of the galenical and other preparations which we have classified as Group *C*, inspection should ordinarily be confined to the records and methods of biological testing, and, when necessary, to the filling and sealing of the containers.
 - (h) That products made abroad should be subject to restrictions similar to those applying to products made in the United Kingdom, and that licences should be granted to approved manufacturers based upon a preliminary inspection of plant, premises and processes, and testing of samples. In addition, steps should be taken to ensure that each consignment attains the standards laid down for similar products of home manufacture.
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THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

AN Ordinary Meeting of the Society was held on Wednesday, May 4, 1921, in the Chemical Society's Rooms, Burlington House. The President, Mr. Alfred Smetham, was in the Chair.

Certificates were read for the first time in favour of Messrs. W. N. Stokoe, B.Sc., A.I.C., and J. F. F. Rowland. Certificates were read for the second time in favour of Messrs. William Ellard Woolcott and Thomas Henry Pope, B.Sc., F.I.C.

Messrs. Percy Nicholas Mould and Walter Joseph Wright, F.I.C., were elected Members of the Society.

The following papers were read: "Detection and Estimation of Illipé Nut Fat used as a Substitute for Cacao Butter," by Francis G. H. Tate, F.I.C., and John W. Pooley, B.Sc., A.I.C.; "Notes and Demonstration on Apparatus for determining Hydrogen Ion Concentration," by G. W. Monier-Williams, O.B.E., M.C., M.A., F.I.C.; "A Note on the 'Oil' of Oats," by Ernest Paul, B.Sc., A.I.C.; and "Estimation of Potassium in presence of Sodium, Magnesium, Sulphates, and Phosphates," by H. Atkinson, B.A., A.I.C.



DETECTION AND ESTIMATION OF ILLIPÉ BUTTER USED AS SUBSTITUTE FOR CACAO BUTTER.

By FRANCIS G. H. TATE, F.I.C., AND JOHN W. POOLEY, B.Sc., A.I.C.

(Read at the Meeting, May 4, 1921.)

THE identification and approximate estimation of certain of the fats used in the cheaper forms of chocolate present little difficulty, and these fats are easily identified by generally accepted methods and data provided in the leading textbooks.

The fat known commercially as illipé butter, however, appears not to have received that consideration which its extensive use and close similarity to cacao butter demand. In the state in which it is usually offered for sale, this fat can be distinguished from cacao butter by the absence of the typical smell of the latter and by its green colour.

It has, however, generally been considered impossible to detect with certainty the presence of illipé butter when mixed, even in appreciable quantity, with cacao

butter, and it became of great importance to investigate the physical and chemical features of these two fats. For this purpose a number of samples were obtained from widely different sources.

The following properties were considered: Specific gravity at 60°/15.5° C., and at 99°/15.5° C., coefficient of expansion, viscosity, melting-point, iodine value (Wijs), refraction $[n]_D^{45}$ (Zeiss), saponification equivalent, mean molecular weight of fatty acids, melting-point of free fatty acids, and "titre" test on fatty acids.

The methods throughout were adopted having in view the fact that the data obtained would be used under the ordinary routine conditions of a laboratory, and that the refined methods sometimes adopted in research would not always be possible.

It was obvious from the figures obtained that the coefficients of expansion were so close as not to form a good basis for comparison. The viscosity was determined at 60° C. in Redwood's instrument. A few readings were taken at 95° C. to establish the "viscosity temperature slopes," but the latter proving to be practically identical for the two fats, this investigation was not pursued.

Melting-Point.—The following method was adopted: A porcelain crucible containing mercury was placed on a wire gauze over a very small flame, the thermometer being suspended therein with the bulb fully immersed. A shaving of fat was placed upon the surface of the mercury, and the temperature raised gradually, at the rate of not more than one degree per minute. The reading was taken when the first signs of transparency were observed on the shaving. Before being finally adopted this method was tested by the work of three different observers, two of whom had no knowledge of the object of the investigation. The results confirmed our opinion that the personal factor could be ignored.

The Saponification Equivalent was determined in the usual manner. It was found, however, that the value of cacao butter and illipé butter covered almost identical ranges, but under the ordinary working conditions of the laboratory consistent results could not be obtained. It was therefore decided to reserve this constant for further investigation if considered desirable.

Fatty Acids.—The fatty acids were prepared by heating 10 grms. of the fat in a porcelain basin with 50 c.c. of alcoholic potassium hydroxide solution (40 grms. per litre) until the resulting soap became pasty. It was then dissolved in hot water, and the solution boiled and acidified with dilute sulphuric acid. The fatty acids were washed by boiling with water until free from sulphuric acid, and then dried and submitted to the following tests:

Melting-Point.—The method adopted for the fats was used.

Mean Molecular Weight.—This was estimated by titration against $\frac{N}{10}$ alkali, phenolphthalein being used as indicator. As might be anticipated, the objection inherent in the saponification test applied equally to this test, and it was accordingly rejected. A summary of the observations is appended:

Cacao Butter.—(12 observations): Minimum reading, 272.6; maximum reading, 296.0; average reading, 280.5; and mean reading, 284.3.

Illipé Butter.—(8 observations): Minimum reading, 269.4; maximum reading 296.7; average reading, 277.6; and mean reading, 282.5.

TABLE A.—CACAO BUTTER.

Specific Gravity at 60° C.	Specific Gravity at 99° C.	Viscosity at 60° C.	Melting-point, ° C.	Iodine Value.	Melting-point of Fatty Acids, ° C.	Refraction [n_D^{40}]
A. 0.8829	0.8581	101.3	30.2	35.2	48.5	1.4568
B. 0.8824	0.8579	100.7	30.6	40.1	48.8	1.4569
C. 0.8823	0.8575	99.1	31.0	35.8	48.1	1.4569
D. 0.8826	0.8575	100.4	31.0	35.0	48.3	1.4569
E. 0.8825	0.8575	99.1	30.7	37.5	48.4	1.4569
F. 0.8826	0.8576	100.3	31.2	36.1	47.7	1.4569
G. 0.8824	0.8572	100.3	30.9	35.6	48.5	1.4570
H. 0.8824	0.8573	99.3	30.9	34.9	48.0	1.4568
I. 0.8824	0.8574	99.9	30.6	36.4	48.5	1.4570
J. 0.8823	0.8573	99.7	31.2	34.6	49.2	1.4570
K. 0.8824	0.8574	99.8	31.1	35.1	48.6	1.4570
L. 0.8825	0.8578	99.0	30.6	35.6	48.5	1.4570
M. 0.8824	0.8574	99.3	30.2	35.7	—	1.4568
N. 0.8823	0.8576	99.6	31.1	36.5	—	1.4570
Average values : 0.8825	0.8575	99.9	30.9	35.9	48.4	1.4569

TABLE B.—ILLIPÉ BUTTER.

Specific Gravity at 60° C.	Specific Gravity at 99° C.	Viscosity at 60° C.	Melting-point, ° C.	Iodine Value.	Melting-point of Fatty Acids, ° C.	Refraction [n_D^{40}]
A. 0.8831	0.8588	102.6	33.8	32.2	52.4	1.4561
B. 0.8835	0.8578	100.7	35.8	32.5	53.4	1.4562
C. 0.8827	0.8569	102.8	30.7	32.4	52.8	1.4568
D. 0.8835	0.8570	102.2	33.8	31.1	52.3	1.4565
E. 0.8827	0.8576	103.0	34.5	29.6	52.5	1.4568
F. 0.8826	0.8573	103.3	34.3	31.3	52.1	1.4568
G. 0.8840	0.8589	—	30.3	30.8	—	1.4564
H. 0.8829	0.8579	105.2	33.0	27.4	—	1.4573
I. 0.8824	0.8576	103.9	34.0	31.0	52.6	1.4568
J. 0.8824	0.8573	104.9	31.7	32.0	52.3	1.4569
K. 0.8824	0.8577	104.7	32.7	30.5	53.0	1.4569
L. 0.8826	0.8578	105.7	34.4	31.6	54.1	1.4571
M. 0.8826	0.8578	104.2	31.0	32.6	52.0	1.4570
N. 0.8820	0.8570	—	33.1	33.2	—	1.4572
O. 0.8823	0.8576	—	33.8	33.4	—	1.4568
P. 0.8826	0.8577	104.6	34.7	31.6	52.5	1.4570
Average values : 0.8826	0.8577	103.7	33.2	31.5	52.8	1.4568

TABLE C.—SUMMARY OF OBSERVATIONS.

	Cacao Butter.			Illipé Butter.		
	Maximum.	Minimum.	Average.	Maximum.	Minimum.	Average.
Specific gravity at 60° C.	0·8830	0·8823	0·8825	0·8840	0·8822	0·8826
Specific gravity at 99° C.	0·8581	0·8572	0·8575	0·8579	0·8569	0·8577
Viscosity... ..	101·3	99·1	99·9	104·9	100·7	103·7
Melting-point	31·2	30·2	30·5	34·7	31·0	33·2
Iodine value	40·1	34·6	39·8	33·4	27·4	31·5
Fatty acids, melting-point	49·2	47·7	49·4	54·1	52·0	52·8
Refraction, $[n]_D^{40}$...	1·4570	1·4568	1·4569	1·4573	1·4561	1·4568

“Titre” Test.—The following modification was adopted: Five c.c. were melted in a test-tube in which a thermometer was suspended without touching the sides of the tube. During the process of cooling, observations of temperature were taken at half-minute intervals, and a time-temperature curve constructed. The “titre”-point was in all cases found to be about 0·5° C. higher than the melting-point. This relation applied equally to the cacao butter and to the illipé butter, and there appeared to be no advantage in adopting this test, which was accordingly rejected.

The observations of the above-mentioned constants were as given in Tables A and B on p. 231, and Table C above.

Consideration of these values indicates that if the average constant could be taken as applicable in all cases, identification and estimation of either fat might be possible, but the variations observed extend the range of each constant, so that for the two fats they often overlap or approximate very closely to each other. It was noticed, however, that if the observed values were compounded so that they could be focussed in one factor this difficulty would be overcome. Further consideration of the values with this object in view demonstrated that all the constants given above would be operative except that for refraction, which is practically identical for both fats. As a general principle it was decided to multiply the various constants together. It was observed that unless all the constants of cacao butter were higher or lower than those for illipé butter, such multiplication would result in one constant nullifying another. To obviate this it was decided that in all cases in which the illipé constant was the lower, the reciprocal should be taken.

The “factors” calculated on this basis were, therefore, the products of the specific gravity at 60° C., the specific gravity at 99° C., the viscosity, the melting-point, the melting-point of the free fatty acids, and the reciprocal of the iodine value.

For twelve individual samples, each of cacao butter and illipé butter, the “factors” thus obtained were as follows:

TABLE D.

Cacao Butter: 3347, 3266, 3252, 3251, 3194, 3191, 3169, 3149, 3130, 3123, 2972, 2839.
Illipé Butter: 4771, 4714, 4652, 4535, 4503, 4487, 4463, 4397, 4279, 4112, 3901 3890.

It will be seen that by this method there are obtained two ranges which are distinct in a measure not to be observed with any individual constant. The respective average "factors" calculated from the average constants of those samples of which complete examination was made are:

Cacao Butter ... 3150 | *Illipé Butter* ... 4403

These figures appeared to afford a reasonable basis for the calculation of proportions in the case of mixtures of cacao butter and illipé butter. It is, of course, evident that such a method, if applied generally and without modification, would be based upon a mathematical fallacy. For example, in the case of the two fats in question, there is an error varying with the relative percentages present and having its maximum in a mixture composed of equal proportions of the two fats.

Table E showing this error was compiled as follows: The constants for the cacao butter and the illipé butter are the accepted averages upon which the above composite factors were based. The values for the mixtures were obtained by numerical interpolation for each constant, and represent the values which should be obtained on average fats if it were possible to eliminate experimental error. The factors given in Column A were obtained by calculation from the interpolated values of the constants by the method described for the two fats under consideration. Column B shows the percentages which would be obtained by referring these "factors" to the two standard "factors."

TABLE E.

	Specific Gravity at 60° C.	Specific Gravity at 99° C.	Viscosity.	Melting-point.	Iodine Value.	Melting-point of Fatty Acids.	A. "Factor."	B. Illipé Butter calculated.
				° C.		° C.		Per Cent.
Illipé butter ...	0.8828	0.8576	103.6	33.5	31.5	52.8	4,403	100.0
90 per cent. Illipé butter	0.8828	0.8576	103.2	33.2	31.9	52.4	4,261	88.7
80	0.8827	0.8576	102.9	33.0	32.4	51.9	4,118	77.3
70	0.8827	0.8576	102.5	32.7	32.8	51.5	3,984	66.6
60	0.8827	0.8576	102.1	32.5	33.3	51.0	3,847	55.8
50	0.88265	0.85755	101.75	32.3	33.7	50.6	3,724	45.8
40	0.8826	0.8575	101.4	31.9	34.1	50.2	3,604	36.2
30	0.8826	0.8575	101.0	31.7	34.6	49.7	3,481	26.4
20	0.8826	0.8575	100.6	31.4	35.0	49.3	3,367	17.3
10	0.8825	0.8575	100.3	31.2	35.5	48.8	3,256	8.5
Cacao butter	0.8825	0.8575	99.9	30.9	35.9	48.4	3,150	0.0

It will be seen that the greatest error is 4.2. This error can, however, be eliminated by applying a correction either to the "factor" obtained or to the percentage calculated from it. The latter procedure is the one we would suggest, and the following table shows the correction to be applied at every even 10 per cent. observed value.

TABLE F.

Observed Percentage of Illipé Butter:	0, 10, 20, 30, 40, 50, 60, 70, 80, 90, 100.
Correction—Add:	0.0, 1.7, 3.0, 3.7, 4.0, 4.2, 3.9, 3.2, 2.2, 1.1, 0.0.

Having regard to the limit of experimental error which must be allowed, it is possible that a result approximating sufficiently closely to the actual value might be obtained by an average addition of 2.1—i.e., half the maximum error—to all observed values.

We prefer, however, to apply the absolute corrections shown in Table F, those applicable to any value other than even intervals of 10 per cent. being easily interpolated.

In order to test the method two series of mixtures were prepared, and each mixture submitted to a full examination. It should be observed that no repetition of work was made, the object being to ascertain how far the method was applicable under ordinary routine conditions. Further, the fats used were not "average" fats obtained by mixing several samples, but were purposely taken at random, and, in the case of the first series, were not previously tested.

The results obtained were as follows :

TABLE G.

Specific Gravity at 60° C.	Specific Gravity at 99° C.	Viscosity at 60° C.	Melting-point.	Iodine Value.	Melting-point of Fatty Acids.	"Factor."	Illipé Butter Present.	Illipé Butter Found.
<i>First Series.</i>								
			°C.		°C.		Per Cent.	Per Cent.
0.8826	0.8575	99.4	31.0	35.0	48.3	3320	10	15.8
0.8827	0.8580	99.8	31.2	33.2	48.8	3466	20	28.6
0.8828	0.8577	100.0	31.5	33.3	49.5	3546	30	35.3
0.8829	0.8577	100.7	30.0	33.3	51.0	3503	40	31.8
0.8830	0.8577	102.0	32.0	32.6	50.8	3853	50	60.1
0.8830	0.8578	102.0	31.4	32.3	50.5	3793	60	55.4
0.8830	0.8579	102.2	32.1	32.2	50.6	3905	70	64.2
0.8830	0.8580	102.8	32.0	31.7	51.0	4010	80	72.0
0.8830	0.8580	103.0	32.3	31.4	52.8	4227	90	87.5
<i>Second Series.</i>								
			°C.		°C.		Per Cent.	Per Cent.
0.8825	0.8577	98.3	32.1	37.3	48.8	3125	0	0.0
0.8825	0.8576	99.3	32.0	36.3	49.0	3245	10	8.9
0.8825	0.8576	99.1	32.5	35.8	50.0	3405	20	23.4
0.8826	0.8576	99.4	32.5	34.7	51.0	3595	30	31.0
0.8827	0.8578	100.2	32.8	34.6	51.2	3682	40	46.4
0.8827	0.8576	101.2	33.0	34.1	51.5	3780	50	54.5
0.8828	0.8578	101.8	32.0	33.5	52.0	3828	60	58.1
0.8827	0.8577	102.0	32.2	33.1	53.0	3983	70	70.0
0.8827	0.8578	102.9	32.8	32.6	53.5	4194	80	85.1
0.8828	0.8577	103.0	33.0	31.9	54.0	4357	90	96.7
0.8829	0.8579	103.4	34.7	31.2	55.0	4790	100	100.0

As a further check, two mixtures were made up containing cacao butter mixed with two illipé fats of different origin, the latter being in about equal proportions, and the following results were obtained :

0.8827	0.8574	100.4	30.9	34.2	49.0	3366	25	19.8
0.8830	0.8576	100.9	32.0	33.2	50.0	3681	50	46.5

It will be seen that the mean deviations were as follows :

TABLE H.

Percentage of Illipé Butter: 10, 20, 25, 30, 40, 50, 60, 70, 80, 90.

Mean Deviation: ... 3.4, 6.0, 5.2, 3.1, 7.3, 6.0, 3.2, 2.9, 6.5, 4.6.

The question which next engaged our attention was the fact that very often the amount available for examination does not exceed 5 grms., whilst complete examination would require at least 130 grms. Certain of the constants which involved the use of only a small quantity of fat were therefore chosen to give a "short" factor calculated in the manner already described. The constants so chosen were: Melting-point, melting-point of the free fatty acids, and the reciprocal of the iodine value. The "factors" so obtained were as follows :

TABLE J.

Cacao Butter: 4437, 4305, 4278, 4258, 4250, 4210, 4192, 4169, 4165, 4123, 3962, 3724.

Illipé Butter: 6119, 5889, 5882, 5875, 5769, 5712, 5685, 5682, 5499, 5182, 5002, 4945.

It will be seen that here, again, the two ranges of values are distinct, the respective factors calculated from the average constants of those samples on which complete examination was made being:

Cacao Butter ... 4166 | *Illipé Butter* ... 5615.

Values calculated on this basis, and corresponding with Table E, show approximately the following correction for every 10 per cent. of illipé butter observed.

TABLE K.

Observed Percentage of Illipé Butter: 10, 20, 30, 40, 50, 60, 70, 80, 90.

Correction—Add: ... 1.5, 2.6, 3.4, 3.9, 3.9, 3.6, 3.3, 2.2, 1.1.

Applying these "factors" and corrections to the values observed for the test mixtures, the following average results are obtained (*cf.* Tables G and H):

Although this "short" factor may be useful where only small quantities of fat are available, we advocate the application of the "long" factor wherever possible. The latter, depending on a large number of observations, is necessarily the more reliable, the possible variation for any constant not affecting so appreciably the final result.

In conclusion, we would point out that we do not regard this investigation as complete. The method is put forward tentatively mainly as a basis for discussion. Further work is proceeding and more data are being accumulated.

TABLE L.

Illipé Butter Present.	Illipé Butter Found.		Mean Deviation.
	First Series.	Second Series.	
Per Cent.	Per Cent.	Per Cent.	
10	8.8	12.2	1.7
20	32.3	28.7	10.5
25	20.7	—	4.3
30	39.3	46.1	12.7
40	33.0	51.4	9.2
50	{ 60.3 49.0 }	60.2	7.1
60			
70	55.1	59.0	2.9
80	64.2	71.7	3.7
80	71.0	85.7	7.3
90	87.8	98.2	5.2

We have to thank the Government Chemist for permission to publish these results, and for facilities for carrying out the work.

GOVERNMENT LABORATORY,
CUSTOMS BRANCH.

DISCUSSION.

Mr. KNAPP pointed out that the general principle of the authors' method might be applicable to other fats, although, of course, there were many pitfalls. He hoped that the authors would give the origin of the cacao butters used, including such details as the type of beans, and he drew attention to the fact that the name "illipé nut" was meaningless, and used in connection with many oleaginous nuts. He supposed that the fat which they had examined was derived from Borneo tallow, but he wondered what would be the interference of other fats, such as shea nut, which might be present. He further drew attention to the fact that equal quantities of given fats would not necessarily give a figure half-way between those of the original components. For example, this was well known to be the case in regard to melting-points. In this connection he asked how long the fats had been kept before the melting-points were taken, as this also affected the melting-point. As this method would be used to detect foreign fats in chocolates, he thought that the fats should first be made into chocolate and then extracted in order to carry out the analysis, as simple mixtures not made into chocolate might not give the same results.

Mr. L. K. BOSELEY observed that it was common practice to grind chocolate in a conche machine continuously for three days and three nights at a temperature of 150° C., and he thought that such treatment might produce some alteration in the analysis of the fats as compared with a simple mixture made in the laboratory.

Mr. E. M. HAWKINS asked upon what principle the authors' system of multiplication and division was based, and raised the question whether the multiplication of certain "factors" might not really magnify the error of experiment.

Mr. E. R. BOLTON thought the method of calculation proposed by the author was distinctly novel, and that the principle might well be applied to other calculations in connection with oil and fat analysis. The method of taking the melting-point appealed to him as being more free from personal error than the standard method. He criticised, however, the procedure of saponifying the fat in a dish, and suggested that a more complete saponification could be achieved in a flask under a condenser, and thus eventually produce purer fatty acids. He asked if all the fats examined were one season's fats, as he had observed a distinct difference from season to season, and he wondered if so far the authors had had any trouble with the fat produced from a species of *dicopsis*, which was a very awkward fat to distinguish when mixed with illipé butter.

Mr. TATE, in replying, said that one particular advantage in their melting-point method lay in the fact that shavings could be taken off the fat and tested without previous melting. It took three days to obtain a constant melting-point after a fat had been melted.

With regard to the question raised by Mr. Knapp and Mr. Boseley, they had extracted the fat direct from test samples of chocolates received from different sources, and the method invariably indicated percentages of illipé butter approximating very closely to the actual. In no case had illipé butter been indicated where not present or missed when used in manufacture.

In connection with the point raised by Mr. Hawkins, Mr. Tate did not agree that the method suffered by any magnification of reasonable experimental error. He explained in detail the process of deduction by which the method was devised. He considered that examination of the values obtained for the mixtures would be found to justify the calculation of the theoretical table.

The authors had examined the fat from two seasons, and the results had been reasonably concordant. Mr. Tate agreed with Mr. Knapp's remarks about the composition of illipé butter. Their work had been done on the fat known commercially by that name, which, in its individual physical and chemical constants, could not be differentiated with certainty from cacao butter. Should there be any fundamental alteration in the character of illipé butter placed on the market, they would immediately detect it, as they were constantly examining fresh samples, and their composite factor would be modified accordingly. At present there was no indication that this would be necessary. If an extraneous fat were present it would be indicated by variation of one or more of the constants.



A NOTE ON THE "OIL" OF OATS.

By ERNEST PAUL, B.Sc., A.I.C.

(Read at the Meeting, May 4, 1921.)

HAVING had occasion to extract some pounds of freshly ground whole oats with petroleum spirit and, with the exception of a paper by Stellwaag,¹ being unable to find in the chemical literature on oils anything very definite as to the characteristics of the oil, advantage was taken of this opportunity for recording them.

The variety used was "Black Tartary," grown on a Fen farm near Peterborough. It yielded 4.32 per cent. of petroleum spirit extract, calculated on the dry sample.

The whole oats were dried to a moisture content of about 4 per cent. at 36° C. in a fruit-drying closet, ground, and extracted by percolation under pressure with hot petroleum spirit of boiling-point 40°–70° C., as long as any appreciable extract was obtained. A bright filtrate was obtained from the spirit extract with the aid of a mixture of kaolin and kieselguhr. On distilling off the solvent and partially drying the residue on a water-bath, it was found that this residue, previously liquid, had become semi-solid owing to the separation of a light brown granular mass, which proved to consist largely, if not entirely, of lecithins. In order to get rid of this, the whole mass was dissolved in dried ether and poured into excess of acetone, the lecithide being thus reprecipitated as a dirty white granular precipitate, which was filtered off by suction and proved to contain nitrogen and phosphorus; when dry and fat-free it only amounted to about 1 per cent. of the total extract.

The oil obtained by evaporation of the filtrate on a water-bath until constant in weight was a clear yellow-green fluid at ordinary temperatures, becoming thick and granular at 10° C., and solidifying completely in twenty-four hours at 5° C.; it had a marked acrid and irritating taste.

The constants of this oil are tabulated below, together with those obtained by Stellwaag on a filtered ether extract not otherwise treated for the separation of lecithins.

König² states that the ether extract of oats consists chiefly of free fatty acids. While this work was in progress a comprehensive article by R. A. Berry³ on oats, including some particulars about the oil, appeared. The main points of interest in this connection are that, according to Berry, the free fatty acids in the oil from freshly ground oatmeal are negligible, but that hydrolysis of the oil occurs to an extent depending upon the time elapsing between the grinding and the extraction, and that the effect of drying is to decrease the rate of hydrolysis, but not to prevent it.

Although no special precautions were taken to guard against hydrolysis or oxidation during extraction, it seems unlikely that free acid to the extent of over one-third of the total extract could have been produced in this way; it seems more probable that, even in the fresh oat, the true fatty matter is associated with a considerable amount of free fatty acid.

¹ Stellwaag, A.: *Landw. Versuchs. Stat.*, 1890, **37**, pp. 135-155.

² König, J.: *Landw. Versuchs. Stat.*, 1874, **17**, p. 1.

³ Berry, R. A.: *J. Agric. Science*, **10**, Part IV., October, 1920.

The analytical figures resemble those of corn (maize) oil. The drying properties of the oil were tested by making films on a ground-glass plate, and it was found that at ordinary temperatures gumming had only commenced in two months; at 99° C. a hard varnish was produced in from two to three days.

On keeping the oil for three months in a corked vessel the acid value was found to be practically unchanged, but the odour had become decidedly rancid.

	Paul.	Stellwaag.
Melting-point ...	Approx. 8° C.	20° C.
Specific gravity at 15°/15° C.	0.925	—
Acid value ...	68.90	70.26
Free fatty acid (as oleic acid)	34.70	35.38
Neutral fat, per cent. ...	64	59.21
Saponification value ...	189.8	192.4
Iodine value (Wijs), per cent.	114.2	—
Unsaponifiable matter, per cent.	1.30	2.65
Refractive index $[n]_D^{20}$	1.4701	—
Total insoluble fatty acids, per cent. ...	93.6	92.76
Acid value of fatty acids ...	196.6	200.8
Mean molecular weight of fatty acids	284.8	279.0
Iodine value of fatty acids (Wijs) ...	127.1	—
Melting-point of fatty acids ...	27.5° C.	—
Refractive index $[n]_D^{40}$...	1.4635	—

The work described above was carried out at the Research Station of the Olympia Agricultural Co., Ltd., The Bury, Offchurch, Leamington, and I have to thank Dr. Crowther, Director of Research, for permission to publish this note.

* * * * *

NOTES.

The Editor desires to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

THE DETECTION OF AGAR-AGAR.

THE detection of agar-agar in foodstuffs, etc., by chemical means offers some difficulty owing to the lack of well-defined reactions and the low chemical activity of this substance.

A method recommended by Macara and others is destruction of the organic matter by oxidation or ignition and examination of the sediment or ash microscopically for the presence of diatoms, which agars usually contain ("Aids to the Analysis of Food and Drugs," Moore and Partridge, Fourth Edition, p. 162. "Food Inspection and Analysis," Leach, Fourth Edition, p. 1002. "Microscopy of Vegetable Foods,"

Winton, Second Edition, p. 320. "Materia Medica," Greenish, Third Edition, p. 217. "Allen's Commercial Organic Analysis," Fourth Edition, Vol. I., p. 438).

This requires extreme care, much skill and experience, and is not always successful. Some samples of agar contain very few diatoms, and the material is only used in small amounts; moreover, when found, diatoms are not always conclusive evidence of the presence of agar. Kieselguhr or diatomaceous earth is sometimes employed as a filtering agent, especially for fruit juices, and the resultant product will often contain diatoms, so that, unless certain diatoms can be definitely identified as being characteristic of agar, the test is unreliable.

Another method recommended is to test the gelatinising power of the solution after removal of the more readily soluble constituents ("Allen's Commercial Organic Analysis," Bolton and Revis, Fourth Edition, Vol. VIII., p. 193).

A modification of this method, when carefully carried out, has been found to give the most reliable evidence of the presence or absence of agar.

For routine testing of fruit products the following procedure is recommended: Fifty grms. of raw-fruit pulp and 100 c.c. of warm water, or, in the case of jams, 50 grms. of jam and 500 c.c. of water at about 50° C., are well mixed and left to stand in a warm place, with occasional stirring until disintegrated, then allowed to settle and filtered through a folded filter. A little alumina cream may be added to assist filtration if sluggish. The insoluble matter is washed with warm water at about the same temperature. This removes sugars and other soluble substances which might interfere with the subsequent test. As a temperature of at least 90° C. is necessary to melt an agar gel, the majority of the latter will be left on the filter. The filter and contents are then transferred to a porcelain or metal vessel and boiled for a few minutes with about 50 c.c. of water and immediately thrown on to a folded filter. The filtrate, on cooling, will set into a gel if it contain any appreciable quantity of agar. If only a small amount, such as 0.1 per cent., be present, it may be necessary to evaporate the filtrate to small bulk before it will gel successfully. The method rarely fails in the presence of agar.

The residue on the filter may be ignited, the ash taken up and gently boiled in a small quantity of dilute hydrochloric acid, the insoluble matter allowed to settle, and the sediment examined microscopically for the presence of diatoms, which, if found in conjunction with a positive gel test, may be considered as additional evidence.

The process can be modified to give a rough approximation of the amount of any agar present by well washing the first filter with warm water and the second filter with boiling water, evaporating the whole of the second filtrate to dryness, and weighing the residue. A pure jam under the same conditions shows only a trace of solid residue.

The amount of agar found should be checked against a solution of agar of approximately the same strength. Agars themselves contain constituents which are soluble in water.

ALBERT E. PARKES:

LEAD IN PEATY WATERS.

The brown colour of certain peaty waters is sometimes so strong as to interfere with the recognition of small traces of lead by treatment with an alkaline sulphide. In such a case the lead may be conveniently recognised and estimated by the following process: Permanganate is added to the water until it is distinctly pink. The water is then rendered alkaline with ammonia and kept for about forty-eight hours, when, unless too much permanganate has been added, the water will be colourless and a precipitate will have formed. This precipitate contains the whole of the lead. It can be collected on a filter, dissolved in a few drops of concentrated hydrochloric

acid, and the resulting solution, after being diluted to a suitable measure, can be tested with alkaline sulphide in the usual way.

This procedure not only produces a colourless solution, but will serve to concentrate the lead without important loss when the proportion of lead in the water is too small for direct examination. The composition of the precipitate has not been studied, but it would appear that peroxide of lead is formed and is carried down either mechanically by manganese dioxide, or, possibly, as a compound with one of the oxides of manganese.

THOMAS TICKLE.



ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Comparison of the Acidity of Goat's, Cow's, and Human Milk. E. W. Schultz and L. R. Chandler. (*J. Biol. Chem.*, 1921, 46, 129-131.)—The acidities were determined by means of standard buffer salt solutions, brom-thymol blue, methyl red, and brom-phenol blue being used as indicators. Fresh goat's milk gave P_{11} values between 6.7 and 6.4, fresh cow's milk from 6.5 to 7.2, and fresh human milk from 6.86 to 7.46. Completely soured goat's milk showed a range of from 4.4 to 3.7, and was considerably more acid than soured cow's milk.

T. J. W.

The Size of Fat Globules in Goat's Milk. E. W. Schultz and L. R. Chandler. (*J. Biol. Chem.*, 1921, 46, 133-134.)—Goat's milk, although possessing a similar fat content to cow's milk, rarely forms a cream layer, owing probably to the small size of the fat globules. Measurements made by the authors by means of dark-ground illumination gave the following results: Below 2μ , 57; 2-4 μ , 34; 4-6 μ , 7; and 6-8 μ , 2 per cent. The fat is thus more finely divided than in the case of cow's and human milk.

T. J. W.

Estimation of Moisture in Milk-Powder. N. Schoorl and S. C. L. Gerritzen. (*Pharm. Weekblad*, 1921, 58, 370-378.)—If milk-powder be dried at either 95° or 103° C. an apparently constant weight is reached in three to four hours, but the results, compared with the actual moisture found by exposure *in vacuo* over phosphorus pentoxide for twenty-four hours at 95° C., are 1.6 to 1.9 per cent. too low; admixture of sand is without influence on the test. Further heating at 95° C. causes gradual loss, but even after twenty-nine hours the correct moisture is not indicated. On prolonged heating at 103° C. the loss in weight fails to reach a limit, and a brown coloration is produced due to decomposition of milk-sugar in presence of phosphates. If milk-powder be heated at 100° C. in air which has been dried by sulphuric acid, the moisture-results after three to four hours are about 0.5 per cent. too low; the correct moisture is indicated only after thirty-six hours' drying. Heating at 110° C. in undried air gives results which are correct within 0.1 to 0.3 per cent. if the time of drying does not exceed two hours, and risk of

decomposition may be avoided by placing the weighing bottles on asbestos instead of directly on the metal shelf of the oven.

W. J. W.

Fatty Acids of Egg-Yolk Lecithin. P. A. Levene and I. P. Roaf. (*J. Biol. Chem.*, 1921, **46**, 193-207.)—The fatty acids were prepared by acid hydrolysis of the salt precipitated by the addition of cadmium chloride to a purified alcoholic solution of the lecithin. These acids were found to consist of a mixture of oleic, palmitic, and stearic acids in molecular proportions. The oleic acid was identified, after separation, by its iodine value and analysis of its hydrogenation product, whilst the two solid fatty acids were identified by their elementary analysis, their melting-points, and their molecular weights. These results tend to indicate the existence of more than one lecithin in egg yolk.

T. J. W.

Electrical Conductivity of Water Extracts of Wheat Flour. C. H. Bailey and F. A. Collatz. (*J. Ind. Eng. Chem.*, 1921, **13**, 319-321.)—Details are given of experiments made to determine the influence of the time and temperature of extraction upon the conductivity of the resulting extracts. This property is slightly increased by extending the time from 15 to 960 minutes, and is more marked in the case of clear flours than in that of patent flours. An increase in temperature also leads to a slight increase in conductivity, the highest values being given at a temperature slightly lower than 60° C. For the comparison of different flours a uniform procedure must be followed, and the following has been adopted: one part of flour is extracted with 10 parts of conductivity water at 25° C. for exactly thirty minutes, with intermittent shaking, whirled in a centrifuge for five minutes, and the clear solution filtered. The conductivity is determined in a modified Freas cell at a temperature of 30° C. in the usual manner. The conductivity of the extracts is apparently due to inorganic salts of phosphoric acid formed by hydrolysis of phytin by the action of phytase, and is proportional to the ash content, thus serving as an index of the grade of the flour.

T. J. W.

Available Carbohydrate in Thrice-Boiled Vegetables. L. O'Reilly and E. H. McCabe. (*J. Biol. Chem.*, 1921, **46**, 83-89.)—The materials investigated were boiled in cheese-cloth bags for three periods of thirty minutes each. The insoluble residues were boiled with water, treated with taka-diastrase, hydrolysed by boiling with dilute hydrochloric acid, and the resulting solution titrated with Benedict's solution. The results obtained indicate that very few vegetables lose the whole of the available carbohydrate present by boiling three times in water. The use of a larger volume of water for boiling ensures a more complete extraction, and the addition of 0.05 to 0.1 per cent. of sodium bicarbonate to the water greatly accelerates the removal of the available carbohydrate. On extracting 100 grms. of vegetable by boiling with three portions of water (2 litres each time), vegetable marrow, lettuce, and celery were completely freed from available carbohydrate, whilst in the case of spinach, asparagus, turnips, beet, and onions, the removal was practically complete. After the same treatment string beans, cauliflower, pumpkin, cabbage, and carrots retain approximately 0.5 per cent. of the carbohydrate.

T. J. W.

Analysis of South Tunisian Palm Wine. A. M. d'Aymeric. (*J. Pharm. Chim.*, 1921, 23, 272-273.)—The "wine" is obtained by puncturing the palm tree, about 7 litres being obtained from a tree per day for a period of more than one month. It is usually consumed in a fresh condition, but readily undergoes fermentation. Analysis of the liquid kept for eight days yielded the following results: Sp. gr., 1.0295; alcohol, 4.5; total acidity (as H_2SO_4), 0.70; glycerol, 2.0; reducing sugars 0.20; gums, 3.0; total solids, 10.5; and ash, 0.20 per cent. W. P. S.

Critical Temperature of Solution of Fats in Mixtures of Ethyl Alcohol and Amyl Alcohol. A. J. J. Vandevelde. (*Bull. Soc. Chim. Belg.*, 1921, 30, 14-16; 58-62.)—Attempts were made to find a mixture of 94 per cent. ethyl alcohol and amyl alcohol which could be used in place of 99.1 per cent. ethyl alcohol in the Crismer method. It was found, however, that a mixture which gave the same value as ethyl alcohol when tested with the standard petroleum spirit yielded low results when used for fats. Further, a mixture which, with beef fat, gave the same value as ethyl alcohol yielded high results when tested against the standard petroleum spirit or coconut oil. The differences appear to be due to the relative solubility of the fats in amyl alcohol (*cf.* Fryer and Weston, *ANALYST*, 1918, 43, 3). W. P. S.

Preserved Edible Fungi. J. Gerum. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1921, 41, 123-126.)—Methods for the canning or bottling of edible fungi (*Boletus edulis*) are described, and it is shown that when they are preserved by sterilising in brine, there is a loss of about 43 per cent. of the nitrogenous food substance if the fungi are subsequently washed and cooked in fresh salt solution. The protein matter so lost is the portion most easily digested. Two samples of the fungi removed from the liquid in which they were preserved had the following composition: water, 92.53 and 93.46; mineral matter, 1.41 and 0.88; organic substances, 6.06 and 5.66; sodium chloride, 1.16 and 0.51; and nitrogen 0.24 and 0.27 per cent.

H. E. C.

Detection of Lactic Acid. L. Hartwig and R. Saar. (*Chem. Zeit.*, 1921, 45, 322.)—Lactic acid may be detected by means of Denigès' reaction, which consists in heating 0.2 c.c. of the solution with 2 c.c. of concentrated sulphuric acid for two minutes in a water-bath, and treating the cooled liquid with one or two drops of a 5 per cent. alcoholic guaiacol solution; in presence of lactic acid, a deep rose-red persistent coloration is obtained, provided that the percentage of the acid in the original solution is not appreciably greater than 0.2. The method is applicable, with the same restriction as regards concentration, to baking powders, which now often contain calcium hydrogen lactate as their acid constituent. The lactic acid may be extracted by stirring about 3 grms. of the baking powder in a mortar with just sufficient water to dissolve it until most of the carbon dioxide has been evolved, then adding a drop of methyl orange solution and running in phosphoric acid solution from a burette until a distinct red tint appears. The liquid is then ground with sand and burnt gypsum to a doughy consistency and left for some hours, the hardened mass being powdered and extracted with ether in a Soxhlet apparatus.

The extract, filtered if necessary, is evaporated in a weighed flask and dried in a steam oven. If lactic acid be present, the residue consists of a viscous, brown syrup, which has an acid taste and fumes in the oven. This is dissolved in water to give a solution of not more than 0.2 per cent. concentration, which is filtered through a hardened filter and tested as above for lactic acid. The reaction is not shown by 0.2 per cent. solutions of formic, acetic, malic, citric, tartaric, benzoic, salicylic, and tannic acids, but is quite distinct with a solution containing 0.022 per cent. of each of these acids and of lactic acid.

T. H. P.

Active Constituents of *Capsella Bursa Pastoris*. H. W. van Urk. (*Pharm. Weekblad*, 1921, 58, 553-556.)—The author failed to detect the presence of alkaloids in a chloroform extract of *Capsella*; very weak reactions obtained with iodine-potassium iodide solution, Mayer's reagent, and tannin were probably due to traces of cholin extracted by the chloroform. A test for glucosides by Bourquelot's method also gave negative results, and Hager's contention (*Pharm. Praxis*, 1, 604) therefore remains unconfirmed.

W. J. W.

Alkaloids of Valerian. A. Goris and C. Vischniac. (*Comptes rend.*, 1921, 172, 1059-1061.)—The authors confirm the existence, in valerian root, of the two alkaloids, chatinine and valerine, discovered by Waliszewski (*Union Pharm.*, 1893, 34, 251); the former is soluble in ether, and the latter insoluble in ether but soluble in chloroform. These alkaloids have but slight physiological activity, and together form only about 0.01 per cent. of the fresh root.

T. H. P.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Variation in the Zinc Content of Vertebrate Animals. G. Bertrand and R. Vladesco. (*Comptes rend.*, 1921, 172, 768-770.)—Examination of a number of animals and fish of various ages shows that the proportion of zinc in the body reaches a maximum at an early stage in the development of the animal, afterwards diminishes to a minimum, and begins to increase in old age. The following amounts of zinc were found in the fresh material: Mice, 2.5 to 4.4; rabbits, 3.1 to 4.9; guinea pigs, 2.3 to 5.6; hens, 3.3 to 31.4; tench, 8.02 (7 months) to 706 (7 years); and herrings 69 to 122 mgrms. per 100 grms.

T. H. P.

Nutritive Value of Lard. J. C. Drummond, J. Golding, S. S. Zilva, and K. H. Coward. (*Biochem. J.*, 1920, 14, 742-753.)—The authors have investigated the question of the known deficiency of lard in the fat-soluble accessory factor (vitamin A). Pigs were fed on diets respectively deficient and rich in vitamins; the pigs were then killed, and the vitamin content of the fat produced under the different conditions of feeding was ascertained by means of feeding experiments with rats. Experiments were also made with pig fat of average character, as used for the manufacture of lard, and with commercial lard obtained from a large firm of bacon-curers. The results of experiments indicate that—(1) When the pig is fed on a diet (*e.g.*, grass) rich in vitamin A, supplies of that factor are stored in the body-

fat. (2) When the diet is deficient in vitamin A (*e.g.*, a diet consisting almost entirely of toppings and whey), the body-fat is deficient in that factor. (3) The process of manufacture of lard on a large scale in this country causes a marked destruction of the vitamin present, probably owing to exposure of the fat to oxygen at a high temperature. It appears, therefore, that the low nutritive value of lard is due to the deficiency in vitamin A of the usual diet of fattening pigs in this country and to destruction of the vitamin during the manufacture of lard.

R. G. P.

Factors Influencing the Nutritive Value of Lard and Lard Substitutes.

J. C. Drummond. (*J. Soc. Chem. Ind.*, 1921, 40, 81-83r.)—The nutritive value of lard cannot be estimated solely in terms of digestibility and coefficients of utilisation, since it depends largely on the amount of the so-called vitamin A present; the diet of the pig is the chief factor controlling the quantity of vitamin in the fat. In the manufacture of lard, loss of vitamin may be prevented by avoiding aeration or close contact with air or oxidising substances at high temperatures. Lard substitutes do not contain appreciable quantities of the vitamin, since this is not usually present in the constituents (cottonseed oil, earthenut oil, oleostearin, etc.). The vitamin content of beef fat is also dependent on the diet of the animal. Hydrogenated fats do not contain the vitamin.

W. P. S.

Anhydrides of Higher Aliphatic Fatty Acids as Food. D. Holde and I.

Tacke. (*Ber.*, 1920, 53B, 1898-1907; *Chem. Abstracts*, 1921, 15, 862.)—The anhydrides of higher fatty acids are readily absorbed by the intestine, and oleic anhydride purified by distillation under reduced pressure has been used as a salad oil, although when used as a frying oil it undergoes some decomposition. The anhydrides of linseed and rape oils and of olein were prepared by heating the glycerides beneath a reflux condenser with a smaller excess of acetic anhydride than in Albitzky's method, and were purified by distillation and treatment with sodium hydroxide. The resulting products were semi-solid at 18° C., rape oil anhydride being partly crystalline, and they melted at about 23° C. They resembled the original glycerides in having a viscous consistency and slight volatility, whilst their refractive indices were about the same as those of the glycerides and higher than those of the free fatty acids.

Use of Edestin in Determining the Proteolytic Activity of Pepsin.

J. F. Brewster. (*J. Biol. Chem.*, 1921, 46, 119-127.)—Edestin was prepared by extracting the fat from hempseed, then extracting the air-dried meal with 5 per cent. sodium chloride solution, and recrystallising the edestin which separated. After filtration the residue was washed with 50 per cent., 95 per cent., and absolute alcohol, and finally with ether, afterwards being air-dried at the ordinary temperature. The proteolytic activity of pepsin was estimated by adding to a series of test-tubes definite increasing amounts of 1 per cent. edestin solution, followed by $\frac{N}{10}$ hydrochloric acid, 10 per cent. sodium chloride solution (which precipitates the edestin), and finally a 1 per cent. dilute hydrochloric acid solution of the pepsin under examination. The tubes were suspended in a constant temperature bath at 37.5° C., and the time noted

which elapsed between the addition of the pepsin solution and the point at which the precipitated edestin was completely liquefied, as shown by the sudden clearing of the contents of the tube. A straight line is obtained by plotting the required time of digestion in minutes against the concentration of substrate, and the relative activities of different pepsins are proportional to $\frac{\text{time of digestion}}{\text{volume of substrate}}$. Initially the activity of one sample of pepsin is determined by the U.S.P. method, and the remainder compared with it by the method described above. The use of edestin as described is based upon the work of Osborne (*J. Amer. Chem. Soc.*, 1894, **16**, 663, 703, 757).

T. J. W.

Effect of Cooking on the Digestibility of Phaseolin. H. C. Waterman and C. O. Johns. (*J. Biol. Chem.*, 1921, **46**, 9-17.)—Feeding experiments made upon rats have shown that the raw proteins of the bean *Phaseolus vulgaris*, added to an otherwise adequate diet, are insufficient for normal growth, but this is achieved if the protein or bean meal be boiled for a short time with distilled water. To investigate this behaviour samples of the raw and boiled phaseolin were digested in a solution containing 0.1 per cent. of pepsin and $\frac{N}{20}$ acid for one and a half hours at 37° C. Sodium carbonate containing trypsin was then added to show, after neutralisation, an excess of both substances of 0.5 per cent., and the digestion continued for two and a half hours. After digestion the enzymic activity was destroyed by heating the liquid to 80° C. for five minutes, the solutions filtered, and duplicate estimations of amino-nitrogen were made upon 10 c.c. portions of the filtrates. The results obtained show that by cooking the phaseolin for five minutes there was a distinct increase in digestibility, the maximum effect being obtained by boiling the phaseolin for three quarters of an hour, when an approximate increase of 30 per cent. amino-nitrogen was produced calculated on that yielded by the uncooked material.

T. J. W.

Vitamins and Yeast Growth. R. J. Williams. (*J. Biol. Chem.*, 1921, **46**, 113-118.)—The vitamin content of various vegetables and yeast was determined by grinding 1 gm. of the dry material with carborundum powder, suspending it in 100 c.c. of water at 60° C. overnight, and filtering the liquid. The amount of filtrate employed in each experiment was such as to give an increase of from 5 to 20 mgrms. of yeast. The results obtained are in good agreement with those obtained by Osborne and Mendel (*J. Biol. Chem.*, 1920, **41**, 451) in rat-feeding experiments. By the yeast-growth method baker's yeast shows a higher vitamin content than does brewer's yeast, but this is reversed in feeding experiments on animals. The extract of each yeast has a greater effect upon the growth of the corresponding yeast than upon that of the other. It is suggested that the antiscorbutic vitamin C may act as a secondary factor in the stimulation of yeast growth.

T. J. W.

Differentiation of Yellow Plant Pigments from the Fat-soluble Vitamin. M. Stephenson. (*Biochem. J.*, 1920, **14**, 715-720.)—Experiments with young rats fed on a diet free from fat-soluble vitamin to which was added (a) crude alcohol-

petroleum spirit extract of dried carrot, (b) pure carotin; and comparative experiments with butter-fat before and after removal of colouring matter by means of birch charcoal, led to the following conclusions: (1) The addition of the crude alcohol-petroleum spirit extract of dried carrot to a fat not containing vitamin (palm-kernel oil was used) confers on it the properties of promoting growth and of protecting the animal from, or curing it of, kerato-malacia. (2) That carotin is not responsible for the above action. (3) That the vitamin content of butter is not affected by the removal of colouring matter by filtration through charcoal.

R. G. P.

Action of Methylene Blue and other Dyes on Living and Dead Yeast.

C. G. Fraser. (*J. Phys. Chem.*, 1920, **24**, 741-748; *Chem. Abstracts*, 1921, **15**, 872.)—On treating suspensions of living and dead yeast with an aqueous solution (0.5 grm. per 100 c.c.) of methylene blue or erythrosin living cells are not stained after several minutes, whereas dead cells are stained within one minute. Congo red, fuchsin, safranin, gentian violet, and methyl green are less suitable dyes for distinguishing between living and dead yeast cells.

Influence of Minute Quantities of Metallic Salts on the Bacteriological Content of Water. E. L. Atkinson and R. C. Frederick. (*J. Royal Naval Medical Service*, April, 1921. *Reprint.*)—Standard solutions of lead (acetate), copper (sulphate), iron (ferric chloride), and zinc (sulphate) were added to four types of water (distilled water and waters low, high, and very high in total solids), so that each sample contained 0.25 or 0.5 part per 100,000 of the respective metals—i.e., amounts within the possible limits of contamination from metal pipes or tanks. One c.c. of an eighteen-hour broth culture of *B. typhosus* was injected into each sample, and also into samples of the waters free from the metals, and these were subcultured at daily intervals for three days into ordinary nutrient broth tubes, which were then kept under extremely favourable conditions, and finally subcultured by the usual routine method. The results, given in tabular form, are contrasted with those obtained in the absence of metallic contamination, and show the very pronounced effect which traces of copper, and sometimes of zinc compounds, may have upon a bacteriological examination of water. It is shown that a water which is unfit for drinking purposes owing to serious contamination with excreta might, if containing minute quantities of metallic compounds, be passed as bacteriologically pure. The results also emphasise the importance of the chemical analysis of any sample of water. The products of contamination of water are readily recognisable by chemical methods, whereas it is admittedly difficult to isolate pathogenic organisms, especially from water contaminated with sewage.

Vitality and Viability of Hæmolytic Streptococci in Water. G. S. Livingston. (*Amer. J. Hygiene*, 1921, **1**, 239-251.)—Streptococci have been found in water contaminated by excretions, the organism of most common occurrence in water contaminated with human faecal matter being *S. fecalis*, a non-hæmolytic variety. Hæmolytic streptococci appear to be rarely present in excretions, and the probability

of their reaching water in this way is slight, although cases of such contamination are on record (Wilson, *War Medicine*, 2, 556). Hæmolytic streptococci are capable of retaining their vitality for periods up to forty days in sterile water, but under more natural conditions the interference of other organisms is an important factor. For example, all streptococci died in less than seven days in pond, ditch, and river water, whilst virulent strains died more rapidly than the least viable of the non-virulent strains, most of them not surviving for twelve hours. The evidence of the experiments described is that virulent hæmolytic streptococci recently isolated from lesions in the human body are poorly adapted to survive storage in water, but that old strains long accustomed to artificial media survive much longer.

Hæmolytic Streptococci from Milk and from Human Lesions. R. C. Salter. (*Amer. J. Hygiene*, 1921, 1, 154-181.)—Hæmolytic streptococci differing in many respects from human pathogenic streptococci occur normally in unboiled and in pasteurised milk, and apparently belong to a homogeneous group. The morphological differences between milk and human hæmolytic streptococci are, however, too slight for purposes of differentiation, but the reaction of milk streptococci in litmus milk is distinctive. Most of the milk species produce acid and cause a reduction of the litmus within twenty-four hours, whereas human pathogenic strains usually produce only a slight acidity, although there are numerous exceptions—*e.g.*, *S. viridans*. Pathogenic hæmolytic streptococci ferment carbohydrates more readily, a constant difference between the two groups being the failure of the milk species to ferment saccharose. They may also be differentiated by their thermal death-rates in milk, the pathogenic streptococci dying rapidly, whilst the milk species die slowly at 60° C. The identification of hæmolytic streptococci should always be confirmed by a test for hæmolysis in a broth culture. The true Beta type produces hæmolysin in broth, whilst other streptococci of very similar type do not. Hæmolytic streptococci resembling the pathogenic type in every respect have been isolated from "certified" milk. They had only a low resistance to heat, and could be destroyed by proper pasteurisation.

A Streptococcus which produces Ropiness in Milk. H. Violle. (*Ann. Inst. Pasteur*, 1921, 35, 218-229.)—A description is given of the streptococcus which produces ropiness in milk; the organism is widely distributed in milk, butter, cheese, etc., and grows rapidly in media containing lactose, maltose, or sucrose; lactic acid being produced. It does not attack lævulose, dextrose, xylose, starch, dextrin, or casein, but it utilises acid amines. The organism develops rapidly at 30° C.; its growth is inhibited at 46° C., and it is destroyed by heating for thirty minutes at 60° C. The streptococcus does not appear to have any injurious effect on human beings.

W. P. S.

Relation between Lactic Acid Production and Bacterial Growth in the Souring of Milk. J. C. Baker, J. D. Drew, and H. J. Conn. (*New York Agric. Exper. Stat., Bul. No. 74*, 1919.)—Different cultures of lactic acid bacteria grow or multiply at different rates; one culture examined by the authors multiplied at a rate

almost in accordance with geometrical progression, whilst with another culture the rate was arithmetical. The rate of acid production per individual cell per hour also varies with the culture, being lower in the case of the organism multiplying at an arithmetical rate than in that which multiplies normally (geometrically). For a vigorous culture, the rate of production of lactic acid lies between 5×10^{-10} and 10×10^{-10} mgrms. per hour. The ratio of total acid produced to the number of organisms present is fairly constant, and is directly proportional to the amount produced per generation by each individual cell. There is a tendency for the acid to increase at a geometrical rate up to the coagulating point; at this point, multiplication of the bacteria appears to cease, and there is a considerable decrease in the rate of acid production. For counting the number of bacteria, the authors consider the direct count method to be preferable to the plate culture method.

W. P. S.

WATER ANALYSIS.

Estimation of Active Carbon Dioxide in Water. I. M. Kolthoff. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1921, 41, 97-112.)—The solubility product of calcium carbonate, and the equilibrium between carbonate, bicarbonate, and carbon dioxide in water are discussed. It is shown that the value of the solubility product obtained by Tillmans and Heublein (*Gesundheitsin.*, 1913, 35, 669) is too high; and the amounts of free carbon dioxide calculated from the equilibrium between calcium carbonate and bicarbonate on the basis of their figure is only correct when the combined carbon dioxide is less than 100 mgrms. per litre. Similarly Tillmans and Heublein's table, from which the active carbon dioxide (*i.e.*, the carbon dioxide which will act on other carbonates) can be calculated, gives erroneous results when the calcium and bicarbonate concentrations are not equivalent. A table is given from which the amount of the active carbon dioxide in a water can be accurately determined when the free carbon dioxide and the calcium content have been estimated. For the estimation of the free carbon dioxide the author adds 10 drops of 1 per cent. phenolphthalein to 100 c.c. of the water in a stoppered flask, and titrates with $\frac{N}{10}$ sodium hydroxide until the faint pink colour persists for five minutes.

H. E. C.

Estimation and Significance of the Hydrogen Ion Concentration in the Examination of Drinking Water. I. M. Kolthoff. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1921, 41, 112-122.)—The use of the hydrogen electrode is not convenient for the estimation of the hydrogen ion concentration in drinking waters, but the colorimetric method gives good results: neutral-red should be used as indicator. The P_H values are found by comparing the colour with that of standard buffer solutions in suitably calibrated glass wedges. A simple method obviating the use of buffer solutions consists in comparing the colour of 10 c.c. of the water, containing 0.1 c.c. of 0.1 per cent. neutral-red solution, with 10 c.c. of standard mixtures of cobalt nitrate (72.8 grms. of crystallised cobalt nitrate in 1 litre of 1 per cent.

hydrochloric acid) and ferric chloride (45.05 grms. $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in 1 litre of 1 per cent. hydrochloric acid). The P_{H} values are as follows:

Mixture.	P_{H} .	Mixture.	P_{H} .
9.0 Co, 1.0 Fe ...	7.0	5.0 Co, 5.0 Fe ...	7.6
7.0 " 3.0 " ...	7.2	4.0 " 6.0 " ...	7.8
5.5 " 4.5 " ...	7.4	3.0 " 7.0 " ...	8.0

The carbon dioxide content of the water may easily be calculated from the P_{H} concentration and the bicarbonate content. The P_{H} value of drinking water is almost always between 7.0 and 8.0, is affected only slightly by the hardness, and is of but little value in assessing the quality of the water. H. E. C.

ORGANIC ANALYSIS.

Guanidine Carbonate as a Standard Alkali. A. H. Dodd. (*J. Soc. Chem. Ind.*, 1921, 40, 89-90r.)—Guanidine carbonate prepared from dicyanodiamide is readily obtained in a pure state by precipitating it from its aqueous solution with alcohol, repeating the precipitation, washing the product with 80 per cent. alcohol, and drying it at 110°C . It is not hygroscopic, and should form a useful substance for standardising acid solutions. W. P. S.

Indicator Properties of Two New Phthalëins. W. Csanyi. (*Zeitsch. Elektrochem.*, 1921, 27, 64-68; *J. Chem. Soc.*, 1921, 120, II., 270.)—The phthalëin prepared from phthalic anhydride and 1 : 2 : 3-xyleneol affords a useful indicator, the change of which ranges from colourless to blue, takes place within the hydrogen ion concentrations, P_{H} 8.9 to 10.2, and is unaffected by excess of alkali or of alcohol. Ortho-anaphtholphthalëin is a less useful indicator than the above, because its colour changes are less sharp, although they take place over the same range of hydrogen ion concentration. Also it is only slightly soluble in water and in alcohol. H. E. C.

Analysis of Liquid and Gaseous Mixtures of Ether, Alcohol, and Water. I. Masson and T. L. McEwan. (*J. Soc. Chem. Ind.*, 1921, 40, 29-32r.)—The method is based on the fact that paraffin, water, ether, and alcohol together form two layers, the upper containing all the paraffin, most of the ether, and a part of the alcohol. By working under standard conditions the increase in weight or volume of the upper layer serves to indicate the amount of ether present, and from the specific gravity of the original liquid and the amount of ether found the amount of alcohol present is estimated by referring to the table of gravities of known mixtures of alcohol, ether, and water determined by the authors. The following is the method of estimating ether in the case of liquids: A separating funnel, containing 200 c.c. of petroleum spirit (sp. gr., $20/4^\circ$, 0.68; b.-pt., $40-75^\circ \text{C}$.), is weighed (to 0.1 gm.); 100 c.c. of the sample are then added and the funnel and contents again weighed. Two hundred c.c. of water, containing 2 per cent. of sulphuric acid to prevent emulsification, are then poured in, and the funnel is shaken vigorously and set aside for a few minutes, with occasional rotation to assist in separation of the two layers. After running off the aqueous layer the gain in weight represents ether, etc.,

absorbed by the petroleum spirit, the gain in weight being about 90 per cent. of the ether present, and from the result the true value is found by reference to curves drawn from the tabulated results of examination of known mixtures. The results are accurate within 1 per cent., except where much water is present. Where only a small quantity of liquid (*e.g.*, 20-30 c.c.) is available, the process is carried out volumetrically in a 100 c.c. burette, the conditions of experiment differing slightly from those recorded above, and the actual content of ether is found by reference to special tables of results obtained by this method with known mixtures. In the case of gaseous mixtures, the gas is first dried by passing it through a drying-tube packed with ignited alumina; the ether and alcohol are then absorbed by two acid-bubblers, containing 10 c.c. and 5 c.c. respectively of pure sulphuric acid (sp. gr., 1.84). The gas taken for analysis should not contain more than 5 grms. of ether; any alcohol absorbed by the alumina should be displaced by a stream of dried air. It is advisable to stand the acid-bubblers in cold water. The gain in weight of the alumina tube gives the amount of water present, and that of the acid-bubblers the alcohol and ether. The contents of the first acid-bubbler are placed in a Mohr-shaped burette, filled to the lowest mark with mercury, and having a 50 c.c. graduated stem and a bulb of not less than 80 c.c. The first bubbler is then washed out with the contents of the second, and the acid layer made up to 20 c.c. with sulphuric acid (sp. gr., 1.84); mercury is then run out and 40 c.c. of paraffin added (sp. gr. at 20/4° C., 0.75; b.pt., 120-140° C.). More mercury is then run out and 50 c.c. of water are carefully introduced into the burette, held in a tilted position, after which the burette is corked and the contents mixed gently while the burette is cooled under the tap. After thorough mixing of the contents by inverting the burette twelve to fifteen times, the liquids are allowed to separate and the gain in volume of the paraffin is read. All readings of the burette are made at the same temperature as indicated by the thermometer attached to the stem of the burette. The gain in volume of the paraffin under these conditions is from 70 to 84 per cent. of the volume of ether absorbed, and the actual amount of ether is obtained by reference to tabulated results obtained by this method with known mixtures.

R. G. P.

Non-Identity of Elæostearic Acid Tetrabromide from Tung Oil with Ordinary Linolic Acid Tetrabromide. B. H. Nicolet. (*J. Amer. Chem. Soc.*, 1921, 43, 938-940.)— α -Elæostearic acid (m.pt., 48° C.) from tung oil yielded, on bromination in glacial acetic acid solution, only 5 per cent. of a solid tetrabromide, m.pt., 115° C.; the corresponding tetrabromide from linolic acid obtained from cottonseed oil melted at 114° C., but when mixed with the α -elæostearic tetrabromide gave a mixture melting at 103° C. Further, ethyl tetrabromo- α -elæostearate (m.pt., 50° C.) was prepared, and its melting-point was found to be lowered by the addition of ethyl tetrabromolinolate. The two tetrabromides are, therefore, not identical, but isomeric, and there is thus no evidence that the two acids are space isomers.

W. P. S.

Determination of the Acetyl Value of Fatty Substances. E. André. (*Comptes rend.*, 1921, 172, 984-986.)—From the saponification values, *S* and *S'*, of a

fat before and after acetylation, it is possible to calculate exactly the corresponding acetyl value. If the acetyl value be denoted by A , it is easily shown that $A = (S' - S) \left(1 + \frac{\lambda S}{1 - \lambda S} \right)$, where λ has the value 0.75. Calculation of the acetyl value from this expression is simpler and more rapid than Lewkowitsch's method for determining it.

T. H. P.

Composition of French Oil of Turpentine. M. Vèzes. (*Comptes rend.*, 1921, 172, 977-980.)—The polarimetric method described by Darmois (Thesis, Paris, 1911) has been applied to the estimation of the proportions of pinene and nopinene in a number of samples of oil of turpentine. Two hundred c.c. of the oil are distilled to five equal fractions of 40 c.c., the specific gravity (d) of each of these being measured at 15° C., and also the rotation (α) of a column 1 cm. in length for the three colours, yellow ($\lambda = 578 \mu\mu$), green (546), and indigo (436), furnished by a mercury vapour lamp and isolated by filtration through suitable glasses. The corresponding values of $[\alpha] = \frac{\alpha}{d}$ are calculated and are plotted as ordinates, with the values of λ as abscissæ. Two points representing, for two colours, each of the five fractions are joined by straight lines, the five lines meeting in a point, C ; straight lines are then drawn between C and the analogous points P and N , corresponding with mixtures of the optical antipodes of pinene and nopinene respectively. By the ordinates of the points where it cuts the vertical lines representing the two colours considered, the line CP defines the rotatory powers for these colours of the pinene in the sample, and similarly CN indicates the rotatory powers of the nopinene present. From the known values of the rotatory powers of pinene and nopinene for one of the colours chosen, it is possible to calculate the proportions of the two hydrocarbons by the law of mixtures. The points P and N lie on the λ -axis, their abscissæ being: for P , 818 and 730.85 for the yellow-green and yellow-indigo pair respectively; and for N , 1042 and 1480.58 respectively. The points C , found experimentally for different samples of genuine oil of turpentine, differ but slightly from one another and also from the point of intersection, C_1 , of the two lines representing, for each pair of colours, the rotatory dispersion of the pinene and nopinene contained in French oil of turpentine. The co-ordinates of C_1 are $\lambda = 740$ and $[\alpha] = 15.10^\circ$ C. for the yellow-green pair and $\lambda = 661.66$ and $[\alpha] = 21.05^\circ$ C. for the yellow-indigo. The divergences of C from C_1 may be due to experimental error or to the existence of varieties of pinene and nopinene differing in rotatory power.

T. H. P.

Estimation of Chlorides in Petroleum. R. R. Matthews. (*J. Ind. Eng. Chem.*, 1921, 13, 325-326.)—The sample is thoroughly mixed, and 500 c.c. are transferred to a graduated 2,000 c.c. stoppered cylinder into which 125 c.c. of acetone are introduced, and the liquids mixed for approximately three minutes. The total volume is then made up to 2,000 c.c. by the addition of water, and the whole mixed for five minutes. The cylinder is allowed to stand until approximately 500 c.c. of the acetone-water mixture have settled out, and about 400 c.c. are

removed by means of a glass siphon, filtered to remove suspended oil, and concentrated, and an aliquot part titrated with $\frac{N}{20}$ silver nitrate solution, potassium chromate being used as indicator. Good agreement is shown between estimations made upon different portions of the same sample of oil. T. J. W.

Estimation of Benzene Hydrocarbons in Coal-Gas. E. Berl, K. Andress, and W. Müller. (*Zeitsch. angew. Chem.*, 1921, **34**, 125-127.)—The adsorptive properties of charcoal are utilized in a method for estimating benzene hydrocarbons in coal-gas. The inherent disadvantages of the dinitrobenzene and paraffin-oil methods are avoided, and the fact that even small amounts can be estimated accurately renders the method superior to that of Deville, in which the minimum amount which can be determined is 26·8 c.c. per cb. m. A layer of charcoal is put into a U-tube, the lower portion of which is widened, and into the open ends of which are placed stop-cocks in communication with inlet and outlet tubes. A current of coal-gas is passed over the charcoal at a velocity of approximately 250 litres per hour. When adsorption is complete, the limbs of the tube are connected with a steam-supply and a condenser and measuring-burette respectively, and the tube is heated in a bath to 110° to 120° C., while a current of steam is passed through it; the hydrocarbon is thus distilled and is measured in the burette. By this method the amount of benzene hydrocarbon in a sample of illuminating gas was found to be 20·2 to 23·9 c.c. per cb. m., whilst the results obtained by the dinitrobenzene and paraffin-oil methods were 16·0 to 17·2, and 9·74 to 10·1 cm. per cb. m. respectively. W. J. W.

Characteristic Reaction of Mercury Fulminate. A Langhans. (*Zeitsch. anal. Chem.*, 1921, **60**, 93-94.)—The test is made as follows: Twenty parts of fulminate are moistened with 5 parts of alcohol, 50 parts of water are added, and the mixture is shaken with 100 parts of 20 per cent. sodium sulphantimoniate solution. A yellow precipitate forms, the colour changing after a time to green and then black. The filtrate obtained from the precipitate is coloured red by nitric acid, and this red colouring matter is soluble in ether. W. P. S.

Reagent for Wood and Vanillin. J. Grüs. (*Ber. Deut. Bot. Ges.*, 1921, **38**, 361-368; *J. Chem. Soc.*, 1921, **120**, II., 284.)—When a wood shaving is dipped in a solution of vanadium pentoxide in dilute phosphoric acid the cell walls gradually assume a yellowish-brown colour. The addition of vanillin to this reagent produces a reddish-brown precipitate, or if the test be applied on a microscope slide reddish-brown crystals may be observed. H. E. C.

Origin, Development, and Value of the Thalleioquin Reaction. W. B. Hart. (*J. Soc. Chem. Ind.*, 1921, **40**, 72-73.)—The green coloration produced when a quinine solution is treated with chlorine and ammonia was first mentioned by H. A. Meeson (*Phil. Mag.*, 1835, 158), and the test has since then been developed in numerous ways by the substitution of bromine for chlorine, by different methods of liberating the halogen, etc. The author, as the result of an investigation of the reaction, finds that excess of bromine and its prolonged action are detrimental to the production of

the coloration, 6 atoms of bromine per molecule of quinine yielding the greatest depth of colour, and with this proportion the reaction may be obtained with a quinine solution of 1 : 200,000. The limit of sensitiveness is 1 : 250,000 in a depth of 2.25 inches of solution. The reaction is governed by so many variables that it is of little, if any, use for quantitative work.

W. P. S.

INORGANIC ANALYSIS.

Identification of Mercury as Cuprous Mercuric Iodide. P. Artmann. (*Zeitsch. anal. Chem.*, 1921, 60, 81-88.)—A bright red coloration is obtained when a drop of a mercury salt solution is added to precipitated cuprous iodide. The reaction is best carried out by coating a piece of filter-paper with freshly-precipitated cuprous iodide, drying the paper at a low temperature, and then placing a drop of the mercury solution on the treated surface. A solution containing 2 mgrms. of mercury per litre will give a coloration with the test. Bismuth salts, easily reducible substances, and excess of acid (greater concentration than $\frac{N}{10}$) must not be present.

W. P. S.

Nitroso R-Salt, a Reagent for the Detection of Cobalt. H. S. van Klooster. (*J. Amer. Chem. Soc.*, 1921, 43, 746-749.)—When R-salt (sodium 2, 3, 6- β -naphtholdisulphonate) is treated with nitrous acid, a nitroso compound is obtained which yields a bright red dye with cobalt salts and renders possible the detection of cobalt in the presence of large quantities of nickel. To prepare the nitroso salt, 35 grms. of R-salt are dissolved in 400 c.c. of water, 10 c.c. of concentrated hydrochloric acid are added, the solution is cooled to 8° C., and a solution of 7.2 grms. of sodium nitrite in 20 c.c. of water is added drop by drop during thirty minutes. The orange-coloured precipitate is collected, washed with a small quantity of cold water and then with alcohol. It is but slightly soluble in cold water, still less in alcohol, and fairly soluble in hot water. To detect cobalt in the presence of nickel and other metals, 2 c.c. of the solution to be tested are treated with 1 gm. of powdered sodium acetate and 2 c.c. of the reagent (0.5 gm. of nitroso R-salt dissolved in 100 c.c. of water); the mixture is boiled, 1 c.c. of concentrated nitric acid is added gradually, and the boiling continued for one minute. A red coloration indicates the presence of cobalt. The colours produced by iron, nickel, copper, etc., are destroyed by the nitric acid.

W. P. S.

Estimation of Iron by the Cupferron Method. G. E. F. Lundell. (*J. Amer. Chem. Soc.*, 1921, 43, 847-851.)—Investigation of the cupferron method for the estimation of iron and the separation of this metal from manganese, showed that iron is precipitated completely by the reagent even from solutions containing as much as 20 per cent. by volume of hydrochloric acid or sulphuric acid; the precipitate is also insoluble in cold dilute hydrochloric acid, but is slightly soluble in dilute ammonia.

W. P. S.

Thallous Ferrieyanide. Estimation of Thallium. V. Cuttica and G. Canneri. (*Gazz. Chim. Ital.*, 1921, 51, I, 169-174.)—The preparation and properties of

thallous ferricyanide are described. The following procedure is recommended in the estimation of thallium as chromate. The liquid containing the precipitated chromate is filtered under pressure through a filter supported by a platinum cone, the precipitate being washed with 80 per cent. alcohol, in which it is practically insoluble, until a drop of the alcohol fails to react for chromic acid. The thallous chromate is then dissolved in dilute sulphuric acid and estimated by addition of potassium iodide and titration with thiosulphate: 3I corresponds with Th_2O . The liquid to be titrated should contain the amount of thallium from about 0.2 grm. of the chromate, and 100 c.c. of 2 *N*-sulphuric acid, diluted to about 400 c.c., should be employed. Two grms. of potassium iodide are added, the end-point of the titration in presence of starch paste being shown by the change from dark green to golden yellow. The method gives rather lower results than the gravimetric method, but is more rapid.

T. H. P.

Preparation of Zirconia from Brazilian Ore and a New Method of Estimation. E. C. Rossiter and P. H. Sanders. (*J. Soc. Chem. Ind.*, 1921, 40, 70-72r.)—The finely ground ore is fused with sodium hydroxide, leached with water, the residue evaporated with hydrochloric acid to dryness and extracted with hot water, and the extract filtered. The dilute solution of the chlorides is treated with sulphur dioxide in slight excess of the amount required to reduce the ferric chloride and heated to boiling, after which sufficient *N*-sulphuric acid is gradually added to replace the chlorine in the zirconium oxychloride; after a short time the whole of the zirconia is deposited as a bulky precipitate of basic sulphate, $5\text{ZrO}_2 \cdot 2\text{SO}_3$. The precipitation, which can be made quantitative, affords a means of separation from iron and alumina, but not from titania. The solution, containing 0.2 grm. of zirconia in 150 c.c., is boiled with 25 c.c. of sulphur dioxide solution until the iron is reduced. It is then neutralised with ammonia, 10 c.c. of sulphur dioxide solution and 2 c.c. of *N*-sulphuric acid added, and the liquid again boiled. The precipitate is filtered off, washed, and dissolved in hydrochloric acid. The solution is evaporated just to dryness, the residue dissolved in water, the precipitation with sulphurous and sulphuric acids repeated, and the precipitate washed and ignited to oxide. The method is not suitable for the separation of large quantities of iron, as the basic zirconium sulphate is slightly soluble in the ammonium salt derived from the acid formed by the reduction of the ferric salt.

W. R. S.

Estimation of Iodic Acid and Silver by Electrometric Titration. W. S. Hendrixson. (*J. Amer. Chem. Soc.*, 1921, 43, 858-866.)—The electrometric titration method is trustworthy for the estimation of iodate; the latter is reduced by the addition of an excess of standardised iodide solution in dilute sulphuric acid, and the excess is titrated with permanganate solution. Iodide may be titrated directly with iodate in dilute sulphuric acid solution by the electrometric method. Hydrochloric acid cannot be used in place of the sulphuric acid. The titrations may, however, be made in the presence of chloride in concentration not exceeding $\frac{N}{10}$, and in the presence of nitric acid of higher concentration if it contains only traces of nitrous acid. Silver may be accurately estimated by electrometric titration, iodide and permanganate solutions being used for the purpose.

W. P. S.

Volumetric Estimation of Sulphuric Acid. C. Pezzi. (*Giorn. Chim. Ind. Appl.*, 1921, 3, 10-11.)—Raschig's method of estimating sulphuric acid volumetrically by precipitating it as benzidine sulphate and titrating the latter, suspended in water, with $\frac{N}{10}$ sodium hydroxide solution at 50° C., is useless in presence of ferric salts, which tenaciously retain occluded benzidine sulphate. The author finds that titration of the precipitate with standard sodium nitrite solution gives results low by only about 0.2-0.3 per cent., even when ferric salts are present. Two grms. of benzidine are dissolved in 750 c.c. of water containing 3 c.c. of hydrochloric acid, the clear solution being made up to a litre; 150 c.c. are sufficient for 0.1 gm. of H_2SO_4 . This solution is added in the cold, with continual stirring, to the sulphate solution containing hydrochloric acid, the precipitate being allowed to settle. The mother-liquor is filtered in small quantities through two filter-papers on a Büchner funnel, the precipitate being afterwards introduced and washed first with the mother-liquor and finally several times with hot water, 15 c.c. in all being used. The precipitate is washed into a beaker, and should be finely divided in the liquid, whilst the papers are destroyed in a small beaker by means of 15 c.c. of concentrated hydrochloric acid, and this solution added to the suspension of the precipitate. The whole liquid is then titrated with $\frac{N}{20}$ sodium nitrite solution at 10-12° C. until, even 15 minutes after the titration is finished, a drop of the liquid colours potassium iodide-starch paper. One c.c. of $\frac{N}{20}$ sodium nitrite corresponds with 0.002452 gm. H_2SO_4 or 0.0024015 gm. SO_4 .
T. H. P.

Detection of Volatile Alkalines in the Presence of Ammonia and of Tertiary Alkalines in the Presence of Primary and Secondary Alkalines. H. E. Woodward and C. L. Alsberg. (*J. Biol. Chem.*, 1921, 46, 1-7.)—The food-stuff is distilled with water, and the distillate received in a slight excess of acid. The acid solution is evaporated to a small volume, made alkaline by the addition of sodium hydroxide, and distilled into about 1 c.c. of 40 per cent. formaldehyde in a test-tube. One c.c. of a solution containing 12 per cent. of potassium bromide and 18 per cent. of mercuric bromide is added, and the test-tube gently warmed. In the presence of over 0.5 mgrm. of amino nitrogen there is produced a white precipitate of mercurous bromide which is insoluble on the addition of more formaldehyde. This reaction is approximately quantitative when employing about 10 c.c. of solution containing 1 c.c. of formalin and between $\frac{1}{100}$ and $\frac{1}{200}$ of the amine. Under these conditions the precipitate obtained is nearly twenty times the weight of the amino nitrogen. To detect tertiary alkalines in the presence of others, the volatile substances are distilled off and collected in a slight excess of dilute acid. The acid solution is evaporated to a small bulk, filtered if necessary, and precipitated by the addition, from a graduated pipette or burette, of Mayer's reagent containing 45 grms. of mercuric iodide and 33 grms. of potassium iodide per 100 c.c., until no more precipitation is observed. Each c.c. of the reagent precipitates 59 mgrms. of trimethylamine. By filtration and distillation of the precipitate with a solution containing sodium hydroxide and sodium sulphide, the trimethylamine may be recovered free from traces of mono- and dimethylamine.
T. J. W.

Decomposition of Nitrous Acid. E. Oliveri-Mandalà. (*Gazz. Chim. Ital.*, 1921, 51, I., 138-140.)—The methods usually employed for the detection of nitric acid in presence of nitrous acid give uncertain results owing to the formation of appreciable traces of nitric acid by partial decomposition of the nitrous acid. The author finds that the action of hydrazoic acid on nitrous acid proceeds quantitatively in accordance with the equation $\text{HNO}_2 + \text{HN}_3 = \text{H}_2\text{O} + \text{N}_2\text{O} + \text{N}_2$, no trace of nitric acid being formed. To test for nitric acid in a solution containing also nitrous acid, the liquid, acidified with acetic acid when the nitrous acid is present as salt, is treated with either a few c.c. of dilute hydrazoic acid or a little of its sodium salt. Since excess of hydrazoic acid interferes with all the colour reactions employed to identify nitrates and nitrites, the liquid is boiled to expel such excess, and a portion of it tested with an acetic acid solution of naphthylamine and sulphanilic acid to ascertain if all the nitrous acid has been destroyed; this being the case, the remainder of the solution is examined for nitric acid.

T. H. P.

Comparison of Scales' Method and the Use of Devarda's Alloy for the Reduction of Nitric Nitrogen. A. P. Harrison. (*J. Biol. Chem.*, 1921, 46, 53-56.)—Two hundred c.c. portions of a sodium nitrate solution containing approximately 0.1 mgrm. of nitric nitrogen per c.c. were measured into Kjeldahl flasks, to one set of which 3 grms. of Devarda's alloy (aluminium 50, copper 45, and zinc 5 per cent.) and 2 c.c. of saturated sodium hydroxide solution were added. A second set was prepared by adding 80 grms. of zinc-copper couple, 5 grms. of sodium chloride, and 1 gm. of magnesium oxide. Both sets were distilled into 50 c.c. of 4 per cent. boric acid solution to a total volume of 200 c.c. One drop of brom-phenol blue was added to each and the liquids titrated by artificial light with $\frac{N}{10}$ sulphuric acid. The results obtained indicate that both methods are equally accurate, but that the Scales' method is more convenient and occupies less time, the same zinc being used repeatedly and recoppered before each estimation.

T. J. W.

PHYSICAL METHODS, APPARATUS, ETC.

Adsorption of Alkalis by Cellulose. I. M. Kolthoff. (*Pharm. Weekblad*, 1921, 58, 46-56.)—Although cellulose does not adsorb sodium and potassium hydroxides in the physical sense, they are taken up by it from solutions, and with concentrations up to 4N the amounts are proportional to the concentration. Thus, from sodium hydroxide solutions of 4N, 2N, N, $\frac{N}{2}$, $\frac{N}{10}$, $\frac{N}{20}$, and $\frac{N}{100}$ strength, undried cellulose removed 3.2, 1.4, 0.65, 0.35, 0.085, 0.04, and 0.11 mol.-equivalents per gm. respectively, and in the equation $\frac{x}{m} = ac$ these results therefore show $a = 0.8$. With dried cellulose the following figures were obtained: from 4N, 2.1; 2N, 1.1; N, 0.58; $\frac{N}{2}$, 0.3; $\frac{N}{10}$, 0.055; and $\frac{N}{20}$, 0.03, giving $a = 0.55$. Between concentrations of 4N and 6N, the amounts taken up are constant and independent of concentration: 3.3, 3.2, and 3.1 for 4N, 5N, and 6N respectively, and chemical combination takes place. Above 6N a sharp increase occurs and amounts again become constant: 5.2 and 5.5 for 7N and 8N; a different combination is formed. Alkali carbonates are not taken up by cellulose.

From barium hydroxide more is taken up than from alkali hydroxides; thus, filter paper took up 0.53, 0.52, 0.28, 0.30, 0.14, and 0.03 mol.-equivalent per grm. from barium hydroxide solutions of 0.29*N*, 0.27*N*, 0.095*N*, 0.087*N*, 0.028*N*, and 0.0084*N* concentration respectively, and with cotton wool the amounts were 0.62, 0.39, 0.22, and 0.14, with 0.3, 0.15, 0.06, and 0.03*N* solutions. The amounts taken up are proportional to the square root of the concentration. Ammonia is taken up only in presence of other alkalis. W. J. W.

Adsorption of Lead and Copper by Cellulose. I. M. Kolthoff. (*Pharm. Weekblad*, 1921, 58, 152-159.)—Neutral solutions of lead salts are not completely adsorbed by cellulose. If an alkali hydroxide containing carbonate be added to the solution, all the lead is removed, but this is due to precipitation of most of the lead, after which the remainder is adsorbed. With very dilute lead solutions, the amount adsorbed by cellulose is dependent on the concentration; in stronger solutions the amounts taken up are proportional to the alkalinity of the ash of the cellulose. Thus, filter paper having an ash-alkalinity equivalent to 1.17 c.c. $\frac{N}{10}$ per grm., adsorbed 1 to 1.4 c.c. $\frac{N}{10}$ per grm. from $\frac{N}{5}$, $\frac{N}{10}$, $\frac{N}{20}$, and $\frac{N}{50}$ solutions of lead acetate, whilst only 0.22 to 0.30 c.c. were adsorbed by filter paper with an ash-alkalinity of 0.22 c.c. $\frac{N}{10}$. In the case of copper salts, adsorption or fixation from neutral solutions is very slight and is not influenced by the alkalinity of the cellulose ash; about 0.1 c.c. $\frac{N}{10}$ per grm. was fixed from solutions of concentrations between $\frac{N}{4}$ and $\frac{N}{40}$. Fixation is greater from ammoniacal copper solutions, and is in inverse ratio to their concentrations. Thus, from solutions of 0.14*N* CuSO₄ in 1.2*N* NH₃, and of 0.07*N*, 0.028*N*, and 0.011*N* CuSO₄ in 0.6*N* NH₃, the amounts fixed were 0.35, 0.37, 0.67, and 0.84, respectively per grm. of paper. A considerable increase in the fixation takes place by addition of sodium hydroxide to the ammoniacal solution: the amounts with 0.14*N* CuSO₄ in 1.2*N* NH₃ and 0.1*N* NaOH, and with 0.07*N* and 0.014*N* CuSO₄ in 0.6*N* NH₃ and 0.1*N* NaOH were 3.12, 5.55, and 5.62 cc. 0.1*N* Cu. per grm. of filter paper. W. J. W.

Adsorption by Asbestos. I. M. Kolthoff. (*Pharm. Weekblad*, 1921, 58, 401-407.)—Asbestos possesses adsorptive properties only when in an impure condition; material which has been purified by treatment with hydrochloric acid is no longer adsorbent. Adsorption varies with different qualities: with five samples, 3.25, 0.65, 0.2, 2.6, and 1.0 c.c. of $\frac{N}{10}$ hydrochloric acid were adsorbed per grm. of asbestos, whilst with another sample, 0.5 c.c. of $\frac{N}{10}$ silver nitrate and 1.37 c.c. of $\frac{N}{10}$ sodium hydroxide were adsorbed. Metals are adsorbed from their neutral solutions. When 1 grm. of asbestos was treated with 50 c.c. of a solution containing 20 mgrms. per litre, and the metal remaining in the solution was estimated colorimetrically after treatment, the following figures were obtained: nickel, 18; copper, 8; iron, 8; silver, 7; and lead 0 mgrms. per litre. The high adsorption of lead may be utilised in its removal from drinking water. Adsorption is found to take place in accordance with the adsorption isotherm: $x/m = ac^{1/n}$. The following values of $1/n$ and a were obtained with various solutions: hydrochloric acid, 0.65 and 4.9; silver nitrate, 0.66 and 0.28; copper sulphate, 0.29 and 0.1; lead nitrate, 0.35 and 0.125; sodium

hydroxide, 0.33 and 0.30; and morphine chloride, 0.53 and 0.2. With higher end concentrations silver is more completely adsorbed than copper or lead, but with lower end concentrations the reverse occurs. Thus, with $\frac{N}{10}$ and $\frac{N}{100}$ silver nitrate, $x/m = 0.075$ and 0.016 ; with 0.1 mol. and 0.01 mol. copper sulphate and 0.05 mol. and 0.005 mol. lead nitrate, $x/m = 0.05$ and 0.028 , and 0.0436 and 0.023 . In addition to being adsorbent, impure asbestos is hygroscopic. For quantitative estimations it must be boiled with hydrochloric acid, and the purified material should not adsorb more than 0.1 c.c. $\frac{N}{10}$ acid per gram.

W. J. W.

Adsorbent Properties of Glass-Wool. I. M. Kolthoff. (*Pharm. Weekblad*, 1921, 58, 463-471.)—Glass-wool is not a true adsorbent, as in contact with water it liberates alkali, which affects the composition of solutions filtered through the material; further, its adsorptive capacity varies with the temperature, and a state of equilibrium is not reached. The alkalinity, calculated as equivalent of $\frac{N}{10}$ hydrochloric in 50 c.c. of water which had been shaken with Jena glass-wool, was 0.05 after five minutes', and 0.5 after ten days', shaking; after boiling for half an hour the alkalinity was 2.6. Previous treatment of the glass-wool with hydrochloric acid slightly increased its action with water, owing to a larger surface being exposed. From very dilute solutions of metallic salts relatively large amounts of the metals are taken up. Estimation of the metal in the residual solution after shaking 60 c.c. (10 mgrms. per litre) for two minutes with 1 gram. of glass-wool at normal temperature and 50°C ., respectively, gave the following results: Lead, 1.5 mgrms. per litre and nil; copper, 2 to 3 mgrms. per litre and nil; zinc, 8 and 1 mgrms. per litre. In each case the adsorbed metal was completely recovered by treating the glass-wool with acetic acid. Investigation of the applicability of the adsorption formula $x/m = ac^{1/n}$ shows that after shaking with hydrochloric acid for one hour, there is no relationship between the end-concentration and the amount absorbed; for values of c of 0.096, 0.0485, 0.02428, and 0.0093, the values for x/m were 0.10, 0.096, 0.072, and 0.070. If the glass-wool be shaken with the acid for five hours to one week, the adsorption equation holds good, and the rate of adsorption is directly proportional to the concentration if there be no interference from diffusion. With end-concentrations of 0.0835–0.0082, the values for α and $1/n$ are 0.89 and 0.35 after five hours' shaking, and 10.0 and 0.89 after one week. Beyond this period, no relationship exists between x/m and c , but the amount which is adsorbed steadily increases. From sodium hydroxide solutions much smaller amounts are adsorbed than from hydrochloric acid; after four hours' shaking, 0.15–0.28 c.c. $\frac{N}{10}$ NaOH were taken up from solutions of $\frac{N}{10}$ to $\frac{N}{100}$ concentration.

From solutions of metallic salts and of alkaloids only small amounts are adsorbed. In the case of silver nitrate solutions, adsorption after four days was 0.25–0.30 c.c. $\frac{N}{10}$; lead solutions undergo a higher adsorption. From quinine hydrochloride only 1.5–2.0 per cent. was taken up. After boiling for fifteen minutes higher results were obtained, the adsorption with $\frac{N}{10}$ silver nitrate, copper sulphate, and quinine hydrochloride, being 2.0, 7.75, and 9.0 c.c. $\frac{N}{10}$ respectively.

W. J. W.

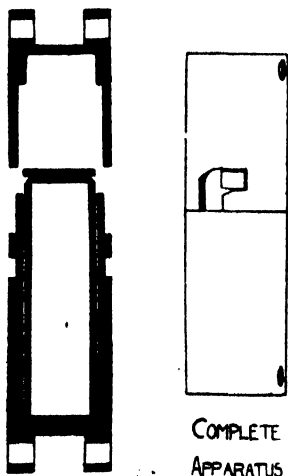
Analysis by means of Reducing Flames; Detection of Traces of Manganese in Presence of Iron or other Substances. J. Meunier. (*Comptes*

rend., 1921, 172, 678-681.)—This method (*Bull. Soc. Chim.*, 1919, 25, 57), which permits of the detection of traces of compounds of lead, manganese, calcium, strontium, potassium, lithium, etc., when other methods fail, is based on the principle that, when an oxide or a salt undergoes reduction on introduction into a flame, the latter exhibits a line spectrum of the corresponding metal. The hydrogen flame gives better results than coal gas, and an apparatus is described by means of which the powdered material is projected into the tube leading the hydrogen to the orifice where it is burnt. Behind the flame is the slit of a spectrograph. The results obtained with oxalate or oxide of iron are given, the number of lines in the spectrum increasing as the exposure of the photographic plate is increased. The presence of a trace of manganese in the iron was readily detected.

T. H. P.

Arsenic Reduction Tube with Electrical Heating. K. Zwicknagl. (*Chem. Zeit.*, 1921, 45, 418.)—In order to obviate the possible danger of hydrogen arsenide passing through the usual form of tube without decomposition on account of insufficient heating of the gas at the centre of the tube, a tube with an internal diameter of 10 mm. is flattened out into a roughly rectangular cross-section by heating and pressing between sheet-asbestos until the walls are about 2 mm. apart. When in use, the flattened part of the tube (15 to 20 cm. in length) is held in a vertical position, and is heated by means of resistance-wire about 0.5 mm. in diameter wound round the tube and separated by and insulated with asbestos-paper—a current of 3 amps. at 120 volts is sufficient to give the necessary temperature; the capillary tube is of the usual dimensions—viz., 1 to 1.5 mm. in diameter.

R. G. P.



COMPLETE
APPARATUS

Apparatus for Sodium Peroxide Fusions. H. J. Hodsmen. (*J. Soc. Chem. Ind.*, 1921, 40, 74T.)—The reaction vessel consists of a thin-walled steel tube closed at the bottom and provided with a well-fitting cap which has so small a surface of contact that a joint cemented by solidified melt can be broken readily or freed by solution in water. The mixture of substance and sodium peroxide is placed in this tube, which is then inserted in an outer casing having a lid held in place by a bayonet catch; the latter, when drawn up, holds the cap of the inner tube in position. The apparatus is supported in an inclined position, and the base is heated by a burner until the charge has fired.

W. P. S.

REVIEWS.

OFFICIAL YEAR-BOOK OF THE SCIENTIFIC AND LEARNED SOCIETIES OF GREAT BRITAIN AND IRELAND. Pp. 351. London: Charles Griffin and Co., Ltd. 1920. Price 15s.

The thirty-seventh annual issue of this Year-Book, which has recently been published, shows a great advance on the previous year in the number of papers contributed on scientific subjects during the session of 1919 to 1920, and is thus an indication of the recovery of the learned societies from one of the effects of the war.

The work is so well known and so permanently established that it is hardly necessary at the present day to emphasise its value to all workers in any branch of science, literature, or art. The complete lists of papers read before the different societies, which are conveniently classified into fourteen groups, enables anyone to learn what has recently been done in the subjects in which he is particularly interested, and in many cases this would not be possible in any other way.

As usual, the book is well printed and on good paper, and has an index which is sufficient for the societies as a whole, but it would be a great advantage if the publishers could see their way to issue every few years a classified subject index.

EDITOR.

COCOA AND CHOCOLATE, THEIR CHEMISTRY AND MANUFACTURE. Second Edition by R. WHYMPER. Pp. 568. London: J. and A. Churchill. 1921. Price 42s. net.

Nine years have elapsed since the appearance of the first edition of this book, which is the standard work in English on the chemistry and manufacture of cocoa and chocolate. Whilst the first edition probably owed more than a little to the standard work in German (Zipperer's "Manufacture of Chocolate"), the 241 pages of new matter in this edition owe nothing to that source of information. The book may conveniently be considered under the three main sections into which it is divided.

The first section, entitled the History, Botany, and Agriculture of Cacao, contains as much information on these subjects as the analyst of cocoa and chocolate could desire. This section has received ample additions in the historical portion, and a separate chapter is now devoted to Statistics (to 1918). One cannot read the interesting but somewhat discursive eighteen pages on the fermentation of cacao without realising how many problems the subject contains for the chemist. The author's whole-hearted approval of the washing of the cacao bean, as practised in Ceylon and Java, is not echoed by other authorities, who have observed that washing away the pulp greatly reduces the toughness of the shell, and renders it brittle and liable to break on handling. The chapter on the characteristics of the principal kinds of commercial cacao bean is excellent, and contains new data of interest to the chemist.

It is the second section, which deals with the Manufacture of Chocolate and Cocoa Powders, which has received the most important and valuable additions, the amount of information being twice that in the former edition. The new material is largely derived from the author's personal experience; it is up to date throughout, and contains much that is of value to the analyst. The few inaccuracies in the first

edition have been corrected, save one referring to theobromine. The author still quotes some results of his which show that large quantities of theobromine are lost on roasting the beans—thus, fifteen minutes at 120° C. show a loss of 0.34 per cent., and eight minutes at 230° C. a loss of 0.86 per cent. The author has evidently been the victim of an inferior method of estimation of theobromine. Were his figures true, the flues leading from roasting machines, in which tons of beans are roasted every day, would soon be clogged with theobromine, and a very valuable by-product thus produced. The new chapter on the Nutritive Value of Cocoa Preparations is unsatisfying, not because of any fault on the author's part, but because the modern work published on this subject is meagre.

The third and concluding section, occupying 177 pages, is of especial interest to the analytical chemist. This deals with the chemistry of cacao and its products, and gives a complete survey of the component parts and the methods of analysis. The author has collected in his book almost all the analytical figures of cacao products that have ever been published, the only notable exception being those of Winton, Silverman, and Bailey. He also gives the range for the various constituents. In the reviewer's opinion the fat in roasted nib is rarely as low as 45 per cent., and the theobromine in cocoa powder never as low as 0.7 per cent. The various analytical methods are set out clearly and fully, and very few processes have escaped mention. A more critical spirit would have been an advantage here; for example, as many as seven methods of estimating the fat are described. The methods of Kreuz and Hughes might with advantage have been replaced by the expeditious process due to S. B. Phillips (*ANALYST*, 1916, xli., 122). Those who, having no knowledge of the subject, consult the book for specific guidance, will find the wealth of material, ancient and modern, confusing, and many of the chapters would profit by a succinct summary of conclusions. Milk chocolate deservedly receives considerable attention, and a chapter has been added, unfortunately not conclusive, on the detection of husk in cocoa, the author's opinion being that "with modern machinery there can be no excuse for the presence of any quantity of husk in a cocoa or chocolate."

On p. 396 theobromine is stated to sublime at 134° C. The correct temperature is about 220° C. Maupy's process does not estimate the mixed alkaloids, as stated on p. 501, but gives theobromine alone.

The text is illustrated by sixteen plates showing the production of the raw product, twenty-nine figures of machinery, and nine photo-micrographs of cocoa and sugar. The photo-micrograph showing crushed cacao nibs is feeble and unrepresentative.

In carefully reading the book only one printer's error was noticed (on p. 515 Method I. should read Method II.). The arrangement of the book is logical throughout, and the printer's work leaves nothing to be desired.

ARTHUR W. KNAPP.

THE EXTRA PHARMACOPŒIA, Vol. II., Seventeenth Edition. By W. H. MARTINDALE, Ph.D., F.C.S., and W. W. WESTCOTT, M.B. Pp. xxxii + 688. London: H. K. Lewis and Co., Ltd. 1921. Price 17s. 6d. net.

The first volume of the seventeenth edition of this work was issued in June, 1920, and has already been reviewed in this journal (*ANALYST*, 1920, Vol. XLV., p. 400).

The subjects included in the present volume are arranged in the same sequence as in Vol. I. They consist mainly of analytical addenda to the *Materia Medica* given in that portion of the work, followed by chapters on Animal Organo-therapy, Bacteriological Notes, and a posological table arranged in alphabetical order.

As in previous editions, we find in this volume a very useful section on the examination of blood, urine, water, and milk, in addition to other physiological and pathological substances. In most instances the methods of analysis described are sound and the deductions made are correct, but a few points call for comment.

In describing the estimation of fat in milk only one method, the Werner-Schmidt process, is given, and the details of procedure in this case are not those usually adopted at the present time.

On p. 450 appears the statement that skimmed and separated milk should contain at least 9 per cent. of milk solids, whereas the Sale of Milk Regulations of 1901, to which the authors refer, have been superseded by the Regulations of 1912, fixing a minimum standard of 8.7 non-fatty solids.

Under water analysis the phenol-sulphonic process is the only one described for the estimation of nitrates, and no reference is made to the other more usual methods.

The present edition retains the chart for the recognition of organic chemical bodies used in therapeutics that has proved its utility in previous issues, and other very useful sections are those on mineral waters, antiseptics, and proprietary medicines. Ninety pages are devoted to Bacteriological and Clinical Notes with reference to special diseases, and the information here recorded is concisely and clearly expressed.

A number of legal enactments and reports on various diseases are referred to, and frequently brief abstracts are given. The methods of bacteriological examination and tests for the detection of specific organisms are described at some length, and the many references to original articles on these subjects show the great trouble taken by the authors to make the book as complete as possible.

The present edition in every respect reaches the high level of excellence attained by its predecessors and should not only find its way to the bookshelves of those specially interested in pharmacology and therapeutics, but should also prove of great value to the large number of analytical chemists who are frequently consulted on matters referred to in its pages.

P. A. ELLIS RICHARDS.

VOLUMETRIC ANALYSIS. By CHARLES. H. HAMPSHIRE, B.Sc., F.I.C. Third Edition. Pp. 120. London: J. and A. Churchill. 1921. Price 7s. 6d. net.

The larger part of this book is devoted to the processes of volumetric analysis and gives a clear presentation of the elementary theory underlying them; there are in addition sections in smaller type dealing with that side of the subject which concerns particularly the pharmaceutical student, for whom the book is primarily intended.

The first six chapters give in detail a variety of estimations based on the use of standard acids and alkalis; the theory of indicators is explained without reference to ionisation. There follow four chapters on reactions involving oxidation and reduction. Two chapters are devoted to precipitation reactions, and finally there is a useful

collection of miscellaneous exercises. Throughout the volume the calculation of results is well illustrated by a series of fully worked numerical examples. The book contains also a short index and a table of the atomic weights of the commoner elements.

As presenting an introductory course in volumetric analysis the small volume can on the whole be recommended; at the same time it is open to criticism on certain points of detail. Need the beginner be burdened with the chemical names and structural formulæ of the dyes used as indicators? If the elementary student is to rely upon his textbook for instruction in manipulation, that instruction should be thoroughly comprehensive. In this book a knowledge of the accurate use of the balance is taken for granted, and no details are given as to the best methods of getting a weighed quantity of solid into solution without loss; on the other hand, the manipulation of the various measuring vessels is described at length. If limitations of space prevent a complete treatment of sources of error, it would surely be better to leave such treatment entirely to the teacher. While the student receives warning on such points as the cleanliness of apparatus and the temperature at which his flasks, etc., hold exactly the right volume, no mention is made of errors of parallax in reading a burette, or of the procedure to be followed if the presence of water in freshly washed apparatus should introduce errors into his results. Omissions such as these are just as fruitful of error as many of those points which are fully dealt with.

A. F. KITCHING.

THE FUNDAMENTAL PROCESSES OF DYE CHEMISTRY. By H. E. FIERZ-DAVID, translated by F. A. Mason. Pp. 235 (45 illustrations, including 19 plates). London: J. and A. Churchill. Price 21s. net.

The title of this book scarcely indicates its technical character, and the word "Manufacture" might well replace "Chemistry." The translator is to be congratulated for introducing to the English-speaking chemist a volume novel in both its manner of presentation and contents. The translation is a very literal one, which is to be welcomed provided the result is a readable volume. This is generally the case, although one notes sentences such as: "For the rest, the index will afford any further information in cases of doubt" (p. 4), and "The development of the dye industry has brought it about . . ." (p. 210). The plates are well produced, but it is unfortunate that the figures are not better adjusted to relate to the adjacent text. Fig. 43 ("Distillation of a liquid of high boiling-point") appears very elementary and quite unnecessary. The publishers are well advised to refrain from the recent practice of producing books on cheap paper and adopting other economies in preference to making a small addition to the cost.

The novel method of inserting as a marginal note the quantities used in a preparation is retained in the translation. Preparations of many important intermediates are given in detail. Objection must be taken to the author's habit of interrupting the description of a preparation by inserting a discussion of the methods which may be used; thus the description of the preparation of naphthalene nitro-trisulphonic acid 1. 3. 6. 8 ceases at the top of p. 14, and is recommenced in the middle of a paragraph on p. 15. The method of stating the amount of an amino-

compound by giving the amount of sodium nitrite in grms. required for its diazotisation is particularly useful, but it should not be introduced in a book of this character without an explanation, as is done on p. 19 ("28 nitrite naphthylamine-trisulphonic acid").

The sections on "Intermediates" and "Dyes" are accompanied by valuable "Notes on Works Technique and Practice" for each preparation. The section on "Technical details" deals with works operations, plant, management, and costing, and a valuable "Analytical section" completes the book.

The author has made an extremely valuable addition to chemical literature. In view of its wide scope, the volume should undoubtedly be in the hands of all interested not only in dyestuffs but also other organic chemicals, either as students, chemists in works, or persons responsible for plant in chemical works.

F. W. ATACK.

RECENT PRACTICE IN THE USE OF SELF-CONTAINED BREATHING APPARATUS. By Lieutenant Rex C. SMART, M.C., R.E. Pp. xiii + 243. London: Charles Griffin and Co., Ltd. 1921. Price 15s.

The subject-matter of this book deals with rescue work in connection with military mining operations, but, as Professor Cadman remarks in a Foreword: "Rescue work in collieries would be conducted more efficiently and with less danger by the application of some, at least, of the rules and regulations, and by the adoption of the standardised system of training which were employed by the military authorities in connection with the rescue operations conducted by the travelling companies of the Royal Engineers during the war." It may be added that, not only in collieries but in many industrial operations where cases of "gassing" are liable to arise, the use of self-contained breathing apparatus, and proper instruction in its use, would prove to be of material benefit.

The author deals in ten sections with (1) the organisation of mine rescue schools, (2) training of personnel, (3) self-contained breathing apparatus, (4) testing and repairing of apparatus, (5) formation of characteristic mine gases in military mining, (6) resuscitation from mine-gas poisoning, (7) organisation of mining companies in mine rescue work, (8) rescue and recovery work in the trenches, (9) use of breathing apparatus in mine warfare, and (10) care of mice and canaries.

The "Proto" type of apparatus, which is described in detail, was the one chiefly used; it has been proved to be in advance of other types, and the extended use to which it was subjected enabled its defects to be detected, and the results of wear and tear to be determined. Doubtless some of its faults were due to the conditions obtaining in military mining operations, and would not arise in civil use where greater care in control and inspection of the apparatus might be anticipated.

The "Salvus" breathing apparatus was used only for inspectional purposes; its wider applicability is disputed in the light of tests carried out with it. A brief description of the "Novita" apparatus for the administration of oxygen in gas-poisoning cases is given.

It is to be regretted that the information in this book has not been presented in a clearer manner; the too frequent use of heavy type, even for minor paragraphs,

confuses the reader and tends to obscure more important items. The paragraph title "Abnormalities and Impurities in Mine Air" on p. 142 is repeated on p. 145. On this page also the incomplete sentence, "Gelignite on detonation 7 per cent. CO, burning 80 per cent. CO," has apparently become detached from its context; whilst on p. 154, a section headed "(b) German Carbon Monoxide Testing Papers" is given undue prominence and should have been inserted at the top of the page after "4 (a) Test paper . . . British method." Notwithstanding these defects, the author is to be congratulated on having collected and assembled a large amount of detail in regard to the use of breathing apparatus, which is a valuable addition to the rather limited information published.

W. J. WRIGHT.



THE INSTITUTE OF METALS.

For the first time since its formation, in 1908, the Institute of Metals will be paying in the autumn a return visit to a provincial city. This meeting will be held in Birmingham, on September 21-23, the first autumn meeting of the Institute having also been held in that city in November, 1908. The arrangements for the meeting are in the hands of the Committee of the Birmingham section of the Institute, of which Dr. H. W. Brownson, M.Sc., is Chairman. The programme will include a reception by the Lord Mayor of Birmingham, visits to the University and works in the neighbourhood, as well as excursions in the locality.

INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY.

BELGIAN BUREAU OF CHEMICAL STANDARDS.

A REPORT has been made to the Belgian Chemical Society (*Bull. Soc. Chim. Belge*, 1921, 30, 41) by M. J. Timmermans upon the Bureau of Chemical Standards, the establishment of which was entrusted to Belgium at the International Chemical Conference (cf. *ANALYST*, 1921, p. 120).

Attention is first directed to the various technical and scientific uses for standard chemical products, and to the difficulty of procuring such products. In Germany, "normal metals" of guaranteed purity can be obtained from the Physikalisch-Technisches Reichsanstalt at Charlottenburg, which has also investigated the methods of purification suitable for technical products and the methods for their analytical control; whilst in the United States an analogous task has been undertaken by the Bureau of Standards at Washington.

The work outlined for the Belgian Bureau falls under three headings: (1) The preparation of pure organic liquids, which at the present time are particularly difficult to obtain. (2) Formation of a depot for pure and standard products prepared in the United States and other countries within the International Chemical Union. (3) The establishment of a centre for the distribution of samples of rare substances, and of information on everything concerning pure products, literature upon the subject, etc.

The work of the Bureau may be assisted by pecuniary help, by supplies of crude material, and by presents of pure chemicals or rare products capable of being used as chemical standards. It is also requested that chemists should send copies of any papers published by them upon subjects cognate to the work of the Bureau.

THE INSTITUTE OF CHEMISTRY.**FINE CHEMICALS, LABORATORY GLASS, AND PORCELAIN.**

THE Council of the Institute of Chemistry, while recognising that the political considerations underlying some of the issues arising from the production, in this country, of laboratory requirements, such as reagents, research chemicals, glass, and porcelain, are outside the scope of the activities of the Institute, nevertheless feel that they are called upon to put before all users of laboratory materials certain definite facts and certain conclusions which may be drawn from those facts.

They have therefore published a memorandum in which it is pointed out that many instances have proved that British manufacturers are capable of producing chemicals in a state of purity fully comparable with that of pre-war supplies from abroad, and that in this matter they feel that it is their duty to emphasise the importance of encouraging home production. They are emphatically of opinion that users of chemicals should make themselves acquainted with what is available as the result of the very substantial progress made by British manufacturers, and, with a view to helping to spread this knowledge, have appointed a special Committee which will deal with questions relating both to reagents and chemicals, and will be prepared to assist chemists to obtain any materials which they may need. All users of such chemicals are strongly urged to make themselves fully acquainted with the circumstances, and to consider the ultimate effect of failing now to aid in building up a stable chemical industry.

Referring to glass apparatus, the Council remark that certain manufacturers have shown ability and readiness to produce the articles required, but that these manufacturers are now under the impression that the support promised to them during the war has not been extended to them in a measure sufficient to make them hopeful of the stability of this part of their industry.

So far as the Council have been able to obtain evidence, complaints regarding glass of recent manufacture, marked with the names of known makers, have been few in number. With the approval of the Board of Trade, the Board of Education, and the Department of Scientific and Industrial Research, the Institute has recently issued a letter to a large number of users urging them to purchase only laboratory glassware which bears the manufacturers' distinctive marks, since without those marks it is impossible to trace the source of any articles which may be the subject of complaints, and to take steps to remedy the faults disclosed.

British-made scientific glassware, equal at least in quality to any hitherto obtainable elsewhere, is forthcoming and at a price which is not unreasonable in the present circumstances having regard to the high cost of materials and production. There is a prospect, moreover, that when once the confidence of the manufacturers is restored and the industry established firmly, prices will compare favourably with those of articles now imported.

The same general considerations apply to the desirability of affording manufacturers of British laboratory porcelain such support as will enable them to complete the final stages of development necessary in order to supply porcelain of at least as high quality as that obtained from abroad. The Council feel that many of the complaints which have been made relate to apparatus of doubtful origin, and for that reason have appointed a Committee which is prepared in the interests alike of users and manufacturers to investigate any complaints which may be brought to their notice.

Summarising the whole position, the Council of the Institute state that they earnestly desire to do all in their power to ensure that chemists shall be able to obtain their professional requirements from home sources, and to aid manufacturers in meeting successfully such requirements.

THE INTERNATIONAL NORMAL WEIGHT FOR THE SACCHARIMETER.

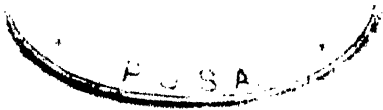
At the present time the normal weight that is in almost universal use for the saccharimeter (or polarimeter, reading the percentage of sugar directly) is 26 grms., this standard having been adopted by the International Commission for Uniform Methods of Sugar Analysis in 1900. On dissolving 26 grms. of pure sucrose in water, and making the liquid up to 100 metric c.c. at 20° C., a solution is obtained which, in a 200 mm. tube, reads exactly 100 scale divisions, corresponding with 34.657 ± 0.023 angular degrees with spectrally purified sodium light. In France a normal weight of 16.29 grms. is largely used.

The proposal, was, however, recently made by Dr. C. A. Browne and other American chemists to adopt a sugar scale having a normal weight of 20 grms., the principal advantages claimed being: (1) It is a compromise between the 26 and 16.29 gm. scales; (2) the results obtained are easily converted into percentages by multiplying by 5; (3) aliquot portions of 50, 25, 20, and 5 c.c. of the 20 per cent. solution represent even gramme quantities; and (4) the specific rotation of sucrose at a concentration of 20 grms. in 100 c.c. (18.62 per cent. by weight) is about the maximum. It was further argued that the factor for conversion into circular degrees, namely $100 = 34.657$, is inaccurate in view of the work of Bates and Jackson published in 1916, and that the present is an opportune time for adopting a new factor and a new standard. French chemists decided to support their American colleagues in adopting the 20 gm. normal weight.

In order to elicit the opinion of British chemists in the matter, a committee was formed consisting of the following: Professor Arthur R. Ling, F.I.C. (who acted as Chairman); Professor Thomas Gray, D.Sc., Ph.D., F.I.C.; L. J. de Whalley, B.Sc., F.I.C.; Hugh Main, B.Sc., and J. P. Ogilvie, A.I.C. (Secretary). This committee drew up a statement of the arguments *pro et contra*, nearly 2,000 copies of which were sent to chemists engaged in the sugar and allied industries, both in this country and in the British colonies.

An analysis of the replies received shows about 72 per cent. of the correspondents to be opposed to the adoption of the new standard. Most of these replies stated as the reason for the decision that the advantages claimed for the proposed new standard were too slight to compensate for the considerable inconvenience, expense, and confusion that would (it was considered) be involved by its adoption. A very frequent additional reason was that, owing to the smaller amount of sample taken for the assay, the accuracy of observation would be diminished. In an addendum to his reply, A. F. Blake, Chief Chemist, Atlantic Sugar Refineries, Ltd., St. John, N.B., Canada, said that he thought the 26-gm. value should be retained regardless of whether Herzfeld's or Bates and Jackson's factor is the correct one, or whether future investigations provide still another, "new instruments being made according to the best factor available at the time, and old ones being controlled and corrected by quartz plates standardised according to the latest factor."

It would therefore appear that British chemists are largely in favour of retaining the present international standard, which in fact is now in almost universal use (excepting in France and in Mauritius).



THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

AN Ordinary Meeting of the Society was held on Wednesday, June 1, 1921, in the Chemical Society's Rooms, Burlington House. The President, Mr. Alfred Smetham, was in the Chair.

A certificate was read for the second time in favour of Mr. W. N. Stokoe, B.Sc., A.I.C.

Messrs. Thomas Henry Pope, B.Sc., F.I.C., and William Ellard Woolcott were elected Members of the Society.

The following papers were read: "The Composition of Egg Powder," by F. F. Beach, F. E. Needs, and Edward Russell, B.Sc., F.I.C.; "The Colorimetric Method of Determining Hydrogen-Ion Concentration: Some Uses in the Analytical Laboratory," by Norman Evers, B.Sc., F.I.C.; "The Estimation of Woody Fibre in Cattle Foods," by F. Robertson Dodd, F.I.C.; "The Spectrometric Examination of Certain Fixed Oils as a Means of Identification," by H. C. T. Gardner; and "The Joint Use of Two Indicators in the Titration of Acids and Bases," by J. L. Lizius, B.Sc., A.I.C.



OBITUARY NOTICE.

BERTRAM BLOUNT.

BERTRAM BLOUNT was born at the end of 1866 in London, and as a child was physically weak, and, indeed, through the whole of his life was never robust, though his indomitable will enabled him to conquer Nature's handicap, and even to attain some success as a cyclist. Educated at King's College School, he passed to King's College, and formed one of the band trained under the late Professor Bloxam; at college he was distinguished by his accuracy and painstaking capacity, as well as by his clearness of thought and expression. At nineteen he joined Mr. W. H. Stanger, a consulting engineer, who held the appointment of Consultant to the Crown Agents or the Colonies, and it is no exaggeration to say that Blount there founded the profession of Engineering Chemist; first as Mr. Stanger's assistant, and then as partner, he forced on engineers and contractors the view that the chemistry of engineering was of prime importance. On Stanger's death, Blount continued the

chemical side of the practice on his own account, and quickly became one of the recognised leaders of his profession ; perhaps he is best known as the leading authority on cement, though his knowledge of electro-chemistry and its practical applications was perhaps equally great, and there was scarcely a branch of inorganic chemistry of which he was not a master.

Few men had a greater power of getting to the root of a problem, and he had the peculiar gift of conveying his thoughts clearly and forcibly, occasionally very forcibly, and he inspired confidence. His contributions to chemical literature were not very numerous ; while still a student he published a paper on calcium borate in the *Journal of the Chemical Society*, followed by one on explosive pyrites ; and his first paper in the *ANALYST* was on a sample of navy green paint. His later communications included "The Estimation of Copper by Ignition in Hydrogen," "The Use of Physics in Analytical Methods," and "The Proposed Standardisation of Methods of Chemical Analysis." With A. G. Bloxam he wrote "Chemistry for Engineers," and was the author of "Practical Electro-Chemistry," and his lecture on "Cement" before the Institute of Chemistry also appeared in book form.

Blount was elected a Fellow of the Institute of Chemistry in 1891, and was a Member of the Council on two occasions, whilst he served as examiner in 1907-1911.

He was always interested in the affairs of our Society. On three occasions he served on the Council, and twice as Vice-President. He was for many years an abstractor for the *ANALYST*, and was subsequently a member of the Publication Committee.

During the war he was strenuous in his demand that the Government should make cotton contraband of war, and in season and out of season he devoted his whole energy to this, and had he been listened to, the war would have been shortened materially and countless lives saved ; the strain on his health probably hastened his end.

Always active, always interesting, never really unkind, though often polemical and sarcastic, he was a true friend and a trusted companion, and his early death on April 9, leaves a gap which can never be filled. H. DROOP RICHMOND.



NOTES ON THE SO-CALLED ACTION OF WATER ON LEAD.

By JOHN C. THRESH, M.D., D.Sc., F.I.C., ETC.

(Read at the Meeting, April 6, 1921.)

THE historical part of this subject was so fully dealt with by Mr. Heap in a paper read before the Society of Chemical Industry (*J. Soc. Chem. Ind.*, 1913, **32**, 771, 811, 847), that anyone interested in this history can easily refer thereto. It will be found that the statements made by different observers are very conflicting, and that there are many anomalies which require explanation.

At my laboratory there are notes, extending over a period of twenty years, on waters which acted upon lead, and when recently a series of waters had to be

examined these records were looked up. Amongst others were notes of the action of samples of distilled water prepared in the laboratory, and some were recorded as "eroding" lead, some as exerting a "solvent" action, some which both "eroded" and "dissolved" lead, and some which apparently did neither. Different fractions from the same distillation had different effects, and it was quite obvious that ammonia-free distilled water must vary considerably in its character. That such was the case was easily demonstrated by examining a number of such waters by the determination of the electric conductivity. The most carefully distilled water had at 20° C. a conductivity of 2, but most gave 3 to 4, and one had a conductivity as high as 14. Certain waters yielded a distillate containing the merest trace of carbon dioxide, others yielded distillates containing considerable quantities, and the degree of oxygenation also differed considerably. It seemed probable, therefore, that these variations accounted for the differences recorded in the action of distilled waters on lead.

The examination of the natural waters above referred to raised other points. One sample was of a river water which, when filtered, contained no lead, whereas, unfiltered and acidulated, it yielded an appreciable colour with hydrogen sulphide. (Lead in large quantities was found in the flocculent sediment in the river, and the drinking of this water by cattle had caused lead poisoning.) Another water, rain water collected in a large concrete-lined tank, and having a long leaden suction pipe to the pump, gave the following perplexing results: Lead per 100 c.c. in water, no acid added, 0.10 mgrm.; after acidifying, 0.25 mgrm.; decanted after standing twenty-four hours, 0.12 mgrm.; after centrifugalisation, 0.10 mgrm.; after filtration through paper, 0.025 mgrm.; and after filtration through Gooch and Pasteur filter, *nil*.

EFFECT OF FILTRATION ON LEAD IN WATER.—To ascertain the effect of filtration, two solutions were made of neutral lead acetate, one with distilled water nearly free from carbon dioxide, and the other with a rain water containing some carbon dioxide and a trace of carbonate. The former solution was quite clear, and the latter faintly turbid. After standing twenty-four hours, successive 50 c.c. were removed by means of a pipette and filtered. (The original solutions contained 0.2 mgrm. of lead in 50 c.c., and the first 50 c.c. of each were examined without filtration.)

					Solution in Distilled Water. Lead found.	Solution in Rain Water. Lead found.
					Mgrm.	Mgrm.
1st	50 c.c.	not filtered	...	...	0.20	0.125
2nd	"	filtered through paper	...	...	0.06	0.04
3rd	"	"	"	...	0.08	0.065
4th	"	"	"	...	0.14	0.10
5th	"	filtered through asbestos	...	...	0.025	0.01
6th	"	"	"	...	0.085	0.03
7th	"	"	"	...	0.13	0.04
8th	"	filtered through porcelain	...	...	0.00	0.00
9th	"	"	"	...	0.025	0.00
10th	"	"	"	...	0.08	0.02

The cylinders in which the waters had stood appeared hazy, as though some deposit was on the glass. This proved to be the case, as, after rinsing, and then adding 50 c.c. of slightly acidified water, both yielded solutions containing lead. The distilled water cylinder yielded 0.375 mgrm., and the rain water 0.30 mgrm. In another experiment with a solution of lead acetate in distilled water (2 mgrms. Pb per 100 c.c.), a minute fragment of sodium was added to give an alkaline reaction. Fifty c.c. of this passed through a filter-paper yielded 0.2 mgrm. of lead, and 50 c.c. passed through a Pasteur filter was free from lead. In another experiment lead foil was placed in a litre of specially distilled water containing the merest trace of carbon dioxide, but saturated with air. The water was contained in a bottle completely filled and stoppered so as to exclude air. After two days the lead was removed, and the liquid poured off was found to contain about 12 mgrms. of lead in 100 c.c. It was diluted so that each 50 c.c. contained 2 mgrms. This liquid was alkaline to naphtholphthalein, and became turbid upon addition of a trace of sodium carbonate. Filtered through the finest filter-paper obtainable, the lead in the filtrate did not exceed 0.005 mgrm., and that passed through a Gooch asbestos filter and a Pasteur filter was quite free from lead. The remainder was passed in successive 50 c.c. through a coarser filter-paper. The first 50 c.c. contained 0.07 mgrm., the second 0.10 mgrm., and the amount passed gradually increased to 0.35 mgrm. in the thirteenth portion, after which the filter practically ceased to pass the water, the pores being choked.

It should here be added that in all cases when the lead solution was rendered faintly acid with acetic or hydrochloric acid, the liquid passed through the filters with its full complement of lead. It is fairly obvious that these neutral or alkaline solutions contain the lead compound in a colloid condition, and that the filtration effects are due to "adsorption."

When pure distilled water containing oxygen in solution acts upon lead, a soluble lead oxide or hydroxide is slowly formed. At first probably there is molecular solution, but molecular aggregates are formed, and the dispersion is then of colloid dimensions; later, larger aggregates may be formed, the dispersion becoming coarser until the liquid appears turbid. When dealing with these minute quantities, forces come into play which are of little importance when considerable masses are being dealt with, as in an ordinary analysis, but which cannot be ignored when dealing with very dilute solutions.

FACTORS AFFECTING THE COLORIMETRIC ESTIMATION OF LEAD.—In estimating quantities of lead from 0.01 to 0.4 parts per 100,000 in various kinds of water, it was observed that the colours produced with hydrogen sulphide varied not merely in depth, but in tint from a reddish-brown to a deep orange, and that frequently the addition of the reagent was rapidly followed by the production of an opalescence which entirely prevented any accurate determination of the lead contents. After a long series of comparative experiments, the conclusion arrived at was that by far the best results were obtained in the absence of a mineral acid (removed by addition of a trace of sodium acetate), and using *N* acetic acid containing as a stabiliser 0.1 per cent. of gold leaf gelatin. The quantity of free acid also affects the depth of colour of the colloidal lead sulphide. The following table shows the effects noted in one series

of experiments in which the quantity of acid used varied. The solution experimented with contained 0.2 mgrm. of lead as oxide or hydroxide in 100 c.c.

Solution: Neutral	With 1% $\frac{N}{10}$ HCl	2% $\frac{N}{10}$ HCl	4% $\frac{N}{10}$ HCl	With 1% $\frac{N}{10}$ acetic acid	2% $\frac{N}{10}$ acetic acid	4% $\frac{N}{10}$ acetic acid	1% N acetic acid
Colour with H_2S : 100	160	185	175	120	175	185	185

The effect due to the variation in quantity of acetic acid above a certain amount is much less than the variation with a mineral acid. The effect of gelatin is even more marked. A natural water containing lead, neutral and not acidified, gave with hydrogen sulphide a coloration of 40; acidified with acetic acid, a coloration of 60; and acidified with acetic acid containing gelatin, a coloration of 100.

The colour produced in the presence of the stabiliser is not only excellent for comparative purposes, but the liquid remains perfectly bright for considerable periods, which is a great advantage when a number of samples are being examined. In the presence of free mineral acid nitrites interfere, but in the quantities ever found in water the effect on the acetic solution is practically *nil*.

INFLUENCE OF HEAT AND AGITATION ON THE SOLUTION OF LEAD.—In making quantitative estimations of the lead taken up by water, it was found that the action was accelerated by heat, but the effect is not such as to necessitate the use of apparatus for maintaining a uniform temperature, the variations at the laboratory temperature being comparatively small, especially compared with other conditions, the chief of which is agitation. The following experiments bring out this point, and also show that the marked effect of agitation is due to the colloid condition of the lead in certain solutions; the colloid lead refuses to diffuse. A tall cylinder (150 c.c.) was filled with distilled water, and a coil of lead-foil placed at the bottom. The upper edge of the lead was opposite the 40 c.c. graduation. After standing at rest for an hour or two, a faint turbidity was noted extending up to this point. After twenty-four hours this had disappeared, but by viewing in different lights there was an obvious difference between the water above the 42 c.c. graduation and below. The upper 50 c.c. were drawn off, and contained no lead; after another twenty-four hours 25 c.c. were drawn off, and contained no lead. After sixty hours another portion of 25 c.c. was pipetted off, and was free from lead. After seventy-two hours 10 c.c. were taken off, and found to contain the merest trace of lead. Then 5 c.c. of remaining liquid were drawn off, and found to contain lead in the proportion of 10.0 mgrms. per 100 c.c., and finally the liquid remaining in the cylinder was acidified with acetic acid, and found to contain 16.5 mgrms. Pb per 100 c.c.

Time.	Agitation.	Lead acted upon by the Water.	Water.
15 minutes	In rotatory machine	2.5 mgrms. per 100 c.c.	Distilled
15 "	Shaken occasionally	2.0 " "	The same water
15 "	At complete rest	1.0 " "	" " "
4 hours	In machine	2.54 " "	A carbonated water
4 "	At rest	1.83 " "	The same carbonated water

In order to obtain uniformity of agitation, a small water-wheel was constructed with a hollow cylindrical axis in which bottles containing water could be placed. With the laboratory water-supply a rate of revolution of between 60 and 70 per minute was easily maintained. This little apparatus has been found most useful. With it the results given on page 273 were obtained.

INFLUENCE OF SURFACE AREA.—The area of lead surface exposed has a marked effect during the first few minutes, but after some hours it is very slight. The unit employed throughout was 25 sq. cms. per 100 c.c., and in all comparative experiments it is necessary to use bottles of the same shape and of approximately the same size. The effect of variation of area of lead exposed is shown in the following tabular results of one series of experiments, made with distilled water containing 0.852 mgrm. of oxygen per 100 c.c.

Unit of Lead Surface per 100 c.c.	Time.	Lead Removed.
Sq. Cm.		Mgrms.
0.5	10 minutes	0.46
1.0	10 "	0.94
1.5	10 "	2.1
2.0	10 "	2.4
0.6	15 " (rotated)	2.0
1.0	15 "	2.5
0.5	6 hours	7.25
1.0	6 "	7.9

The way in which the lead surface is cleaned has a pronounced effect upon the rate at which action commences and continues for about the first hour, but afterwards the effect is only slightly marked. Lead which has been dipped in dilute acid and then polished with a dry cloth has the greatest initial velocity, and the same lead dipped into $\frac{N}{100}$ sodium carbonate solution after treatment with $\frac{N}{10}$ hydrochloric acid had the least initial velocity, but the results were very variable. After numerous experiments, it was concluded that the most uniform results were obtained by using lead which had first been well rubbed with finely-powdered pumice and then polished with clean dry linen.

ACTION OF DISTILLED WATER.—The factors likely to affect the results having been studied, experiments were conducted with distilled water, and after many failures an apparatus was devised for distilling water and collecting the distillate without the access of air, and thus obtaining a water free from dissolved gases, especially oxygen and carbon dioxide. When such a water is obtained and the unit of lead introduced through an atmosphere free from oxygen and carbon dioxide, and the containing vessel is sealed, the water remains clear and the metal bright; and when the water is poured off quickly and tested, no trace of lead can be found in solution.

If to such a sample of water air deprived of carbon dioxide be admitted, the liquid remains clear for two or three days, after which a deposit forms on the bottom of the bottle, and when shaken the deposit exhibits a crystalline character. Under

the microscope it is seen to be very minute and acicular, single and in tufts. It settles quickly and the liquid is bright. If now ordinary air be permitted to gain entrance, the liquid soon becomes turbid from the absorption of carbon dioxide. The solution is alkaline to phenolphthalein and naphtholphthalein, and precipitates with the slightest trace of a solution of a carbonate.

Obviously this is a solution of lead oxide or hydroxide, and the saturated solution contains about 20 mgrms. of lead in 100 c.c. Apparently the amount varies from 19.5 to 20.6 mgrms., and this may be regarded as the solubility of this compound at 8° to 12° C. With free access of carbon-dioxide-free air, the formation of the oxide or hydroxide continues, but when air is excluded, the action stops when the available oxygen is used up. This applies also to distilled water to which any acid has been added. If the acid forms a soluble lead salt, this remains in solution, whilst if the acid forms a practically insoluble lead salt, this insoluble salt is deposited as soon as the water has become saturated therewith. Carbonic acid appears to form the most insoluble salts, followed by silicic acid, then, at a distance, by phosphoric, sulphuric acid, etc. So far as the solvent action of water on lead is concerned, carbonic and silicic acids are the only ones to be considered. When the action between oxygen-laden but otherwise pure distilled water and lead takes place in stoppered bottles, quite full and protected from the access of air, it is always found: (1) That the oxygen taken up by the lead corresponds to the lead which has entered into solution as estimated by hydrogen sulphide. (2) That the loss of weight suffered by the metal is very approximately a measure of the amount of lead which has been oxidised. (3) That the amount of lead dissolved can be estimated with fair approximation by titrating the solution with $\frac{N}{10}$ acid, using methyl orange as the indicator.

(1) This requires that the oxygen contained in the water at the commencement and end of the experiment should be accurately determined, and Winkler's method has been used (save in certain cases where experiments were being made with nitrited waters, when my process was utilised). The following are the results of a series of experiments, commencing with water exhausted as far as possible by an air-pump, and concluding with water through which pure oxygen gas had been passed:

	Lead, Mgrms. per 100 c.c.															
	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.	12.	13.	14.	15.	16. Aver.
Estimated from loss of O (1 mgrm. = 12.04 Pb)	0.0	1.42	2.2	2.7	4.1	7.4	7.3	7.25	9.75	11.1	12.3	14.9	20.7	21.6	35.0	42.8
By means of H ₂ S ...	0.03	1.55	2.1	2.5	4.25	7.3	7.35	8.0	9.4	11.0	12.0	15.6	19.0	22.0	35.3	42.0

The above results show that the amount of lead chemically acted upon is determined by the amount of oxygen taken out of solution, and that the extent of this action can be accurately gauged by the amount of oxygen lost.

(2) This is more troublesome, and is of no practical use. Save with pure distilled water or acidified water the results are useless, as insoluble salts deposit upon the lead and vitiate the results. The following experiments were made with distilled water containing the merest trace of carbon dioxide:

	1.	2.	3.	4.	5.	6.	7.
Weight lost by lead foil (mgrms.) ...	6.2	7.5	9.0	13.2	14.0	14.3	15.0
Lead found in solution and suspension (mgrms.) ...	7.1	8.8	8.45	13.45	13.75	14.5	15.0

(3) This is not of much utility, since the presence of lead salts in solution interferes with the delicacy of the end reaction, but it is worthy of notice that in distilled

waters containing only oxygen and carbon dioxide the amount of lead taken up can be approximately estimated in this way. The attempts to estimate the relative proportion of oxide and carbonate by using phenolphthalein as well as methyl orange all ended in failure.

With distilled water the velocity of the reaction between the oxygen and the lead appears to depend upon the amount of oxygen present, and the same remark applies in the presence of any free acid forming soluble lead compounds, but in the presence of acid the velocity is greater in the earlier stages. For example:

Time.	Lead acted upon—	
	Neutral Distilled Water.	Distilled Water + 4 per Cent. $\frac{N}{10}$ HCl.
	Mgrms.	Mgrms.
$\frac{1}{2}$ hour	1.65	3.1
3 hours	4.7	8.0
5 "	7.1	9.7
24 "	11.0	12.4
48 "	12.2	13.0

RATE OF UTILISATION OF OXYGEN.—The following gives in a little more detail information concerning a series of experiments showing the rate at which oxygen is taken up, and that action ceases when all the oxygen has been used. The bottles used all held approximately 160 c.c., and were completely filled so as to exclude air. After standing the requisite time, 5 c.c. of water were taken from the bottle, the solutions of manganese chloride added (3 c.c.), and some of the original water added again to fill the bottle. In the 5 c.c., diluted to 50 c.c., 100 c.c., or more according to circumstances, the lead was determined, and the residual oxygen was determined in the remainder of the water. The correction for oxygen in the water and chemicals added was usually so small as to be negligible. In many cases duplicate bottles of water were put on, so that the lead could be estimated in one and the residual oxygen in the other. The discrepancies which occurred when this method was used led to the discovery of the marked effect of unequal agitation.

Record of experiments made November 19 and 20, 1920. Temperature, 13° C. Oxygen in 100 c.c. of the distilled water, 1.030 mgrms.; free CO₂, 0.33 mgrm.:

Time.	Oxygen Left.	Oxygen Lost.	Lead Equivalent.	Lead found by H ₂ S.
	Mgrm.	Mgrms.	Mgrms.	Mgrms.
$\frac{1}{4}$ hour	0.95	0.08	1.03	0.6
$\frac{1}{2}$ "	0.90	0.13	1.68	1.66
1 "	0.80	0.23	2.98	2.8
2 hours	0.66	0.37	4.79	4.55
3 "	0.54	0.49	6.34	6.1
6 "	0.44	0.59	7.6	7.1
21 "	0.18	0.85	11.0	11.1
72 "	0.03	1.00	12.95	13.6

EFFECT OF CARBON DIOXIDE.—Carbon dioxide produces very insoluble lead salts; yet it is capable of dissolving a small quantity of lead when it is present in unusual amounts. It is a most important factor in the action of water on lead, since the so-called "erosive" action is entirely due to it. It does not prevent the action of the oxygen dissolved in the water, but it enters into combination with the oxide formed. If the amount of carbon dioxide does not greatly exceed 1 part per 100,000 of water, when lead is inserted action is distinctly visible within a few minutes. The formation of an insoluble lead carbonate can be seen at the surface of the metal, and this glides down and forms a turbid layer at the bottom of the bottle. As the action proceeds, the turbid layer increases in thickness until it just covers the upper surface of the lead. After this, if unshaken, the suspended matter slowly deposits and the water again becomes clear.

When the carbon dioxide exceeds 1·5 parts per 100,000 this turbidity is not produced, the water remains perfectly clear and bright, some lead is taken into solution if sufficient carbon dioxide be present, but a very insoluble carbonate is deposited on the metal, which assumes a dark grey colour. The amount of lead acted upon can be easily ascertained from the oxygen loss. It is very difficult in many cases to determine it directly by hydrogen sulphide, since a considerable amount of strong acid must be added to dissolve the deposit, and if much oxygen is left in the water the action of this acid has to be allowed for. When action has proceeded until nearly all the dissolved oxygen has been used up, the determination is easy, but when much oxygen remains a correction must be made for the amount of lead affected by the acid added. This is best done by taking duplicate samples, which are prepared and kept under identical conditions, and at the expiration of the required time one bottle is used for estimating the residual oxygen, and into the second is introduced 1 per cent. of 99 per cent. acetic acid and the bottle shaken until all the deposit is dissolved off the lead. This usually takes two to five minutes. Now 5 c.c. of the acid water are removed for the determination of the lead by means of hydrogen sulphide, and the remainder is used for the estimation of the residual oxygen. The difference between the two oxygen determinations is the measure of the lead acted upon by the acid, and affords the necessary correction. The following table is explanatory :

Time.	Oxygen in 100 c.c. of Water used.	Oxygen Lost.	Lead Equivalent.	Lead found by H ₂ S.
	Mgrms.	Mgrm.	Mgrms.	Mgrms.
3½ days	1·103	0·52	6·75	—
3½ "	1·103	0·63	8·1	8·0 — 1·35 = 6·65
4 "	1·106	0·21	2·75	4·0 — 1·7 = 2·3
6 "	1·106	0·495	6·3	8·4 — 1·6 = 6·8
7 "	0·515	0·135	1·75	3·15 — 1·6 = 1·55

In the following table the results obtained with oxygenated water containing various quantities of free carbon dioxide are recorded. In the cases where the free

carbon dioxide was less than 1 part per 100,000 the liquid was turbid; where it contained 1.15 parts it was dull; in all the others the water remained clear.

Oxygen in Original Water per 100 c.c.	Free Carbon Dioxide. Parts per 100,000.	Total Lead acted upon calculated from O lost and by H ₂ S.	Lead in Clear poured-off Liquid.	Lead in Filtrate.	
				Pasteur.	Paper.
Mgrms.		Mgrms.	Mgrms.	Mgrm.	Mgrm.
0.70	0.75	7.0	0.20	0.00	—
1.053	1.15	11.65	0.275	—	0.03
1.053	2.65	12.7	0.25	0.03	—
1.031	3.0	13.3	0.30	0.00	—
1.045	4.0	10.7	0.40	0.00	—
1.045	7.9	10.9	1.9	0.00	—
1.045	14.3	9.45	2.85	0.00	0.60

INFLUENCE OF CARBONATES.—When bicarbonates are present action is retarded, and calcium carbonate in carbon dioxide solution has a much greater retarding action than the sodium salt. Of the hundreds of experiments made, a few only can be quoted here. These show that when small quantities of carbonates only are present, the lead when immersed in the water causes more or less turbidity, but when the amount of calcium carbonate present exceeds about 1 part per 100,000 the water remains clear. A larger quantity of sodium bicarbonate is usually necessary to produce this effect.

Time.	Amount of Salt Present, Calculated on Bicarbonate.	Free CO ₂ .	Oxygen Lost.	Lead Equivalent.	Lead by H ₂ S.	Notes.
1 day	(CaC ₂ O ₅) 0.4	0.0	0.51	6.6	—	Turbid. No lead in solution.
"	" 0.9	0.0	0.18	2.3	—	Less turbid. Ditto
"	" 1.35	0.0	0.20	2.6	—	Clear and bright. Ditto
2 days	" 0.5	0.15	0.73	9.45	10.5	Turbid. Trace Pb in solution
"	" 1.0	0.3	—	—	4.8	Clear and bright. No Pb in sol.
"	" 1.45	0.45	0.59	7.65	8.0	" " "
"	" 1.7	0.5	0.22	2.85	2.85	" " "
4 days	" 19.0	0.55	0.22	2.85	2.8	" " "
2 days	(NaHCO ₃) 0.5	0.0	0.77	10.2	10.15	Turbid. No Pb in solution
"	" 1.0	0.0	0.44	5.7	—	Clear and bright. Ditto
"	" 2.0	0.0	0.36	4.65	—	" " "
"	" 5.0	0.0	—	—	—	" " "

When water containing carbonate is kept in constant motion whilst in contact with lead, the water becomes turbid, or at least dull, and if poured off and acidified lead is found to be present. The following experimental results show how markedly the results may be affected by agitation. The water used was a pure chalk water of over 20° hardness.

Time.	Agitation.	Oxygen Present.	Free CO ₂ .	O used.	Lead Equivalent.	Notes.
3 hours	Rotated	1.030	0.0	0.10	1.3	Water dull, but does not deposit
24 hours	At rest	1.030	0.0	0.363	4.7	Water bright. No lead in poured-off liquid

The rotated water gave the following results :

- 50 c.c. poured off and treated with hydrogen sulphide without acidification : lead absent.
 50 c.c. poured off and treated with hydrogen sulphide after acidification : 0.32 mgrm. Pb per 100 c.c.
 50 c.c. poured off and filtered through paper after acidification : 0.10 mgrm. Pb per 100 c.c.

GENERAL CONCLUSIONS.—The effect of numerous salts has been studied, but the results must be reserved for another occasion. The experiments above recorded prove that water has no action whatever on lead ; that it is the foreign matters dissolved in water which affect the metal, the oxygen combining with it to form a soluble oxide or hydroxide, which remains in solution (to the point of saturation) in the absence of other substances capable of combining with it. This solution is of a colloidal nature and of such coarse degree of dispersion or so liable to adsorption that it will not pass through a Pasteur filter, and may not even pass a fine paper filter. In the presence of acids forming soluble lead salts, such salts are formed and will pass through any filter. If the acid forms insoluble salts, these are thrown down, and may be deposited on the metal in such a way as to prevent any further action. Any soluble salts present in the water may likewise act upon the lead hydroxide. Water is merely an inert medium, by means of which these foreign substances are enabled to act upon each other, the water itself taking no part other than that of a solvent.

THE COMPOSITION OF EGG POWDER.

By F. F. BEACH, M.A., F.I.C., F. E. NEEDS, A.I.C., AND EDWARD RUSSELL, B.Sc., F.I.C.

(Read at the Meeting, June 1, 1921.)

THE examination of a substance bearing the name "egg powder" was occasionally undertaken in the City Laboratories, Bristol, and it was generally recognised that it was similar to baking powder, as the table on p. 282, taken from two consecutive years, will show.

Rice starch was the common organic constituent, and the only real difference between these substances was one of colour, effected by the introduction into the

Liquid Egg. 1.	Dried Egg. 2.	Dried Eggs. 3.	Dried Eggs. 4.	Egg Powder. 5.	Egg Powder (Guaranteed to contain Eggs). 6.	Egg Substitute—		Egg Powder. 9.
Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	7.	8.	Per Cent.
Loss at 100° C. (moisture, etc.)	72.8	9.05	8.0					14.5
Ash	0.95	3.17	3.24	7.63	12.72	23.3	8.66	25.16
Calcium sulphate ...				0.22	0.62	1.35	0.21	0.79
CaSO ₄ in ash ...				2.9	4.9	5.8	2.4	3.1
Ethereal extract ...	13.65	40.09	39.07	0.34	0.99	0.19	0.30	0.30
Refraction (Zeiss) at 35° C.	67.5	64.0	67.0					
Nitrogen	6.97	7.04	7.45	0.96	0.97	0.92	0.55	0.74
Protein	43.5	44.0	46.5	6.02	6.06	5.75	3.44	4.62
Organic phosphorus (P ₂ O ₅)	1.28	1.20	1.27	0.0095	0.025	0.0043	0.0088	0.016
Arsenic (grains per pound)				0.00	1.30	0.00	0.10	1.25
Cold water extract				Colourless	Yellow	Deep yellow	Light yellow	Yellow
Tartrates				Trace	Trace	+	+	+
Insoluble matter, phosphates				Rice.	Rice. Potato	Rice and some	Potato. Rice.	Rice
Microscope ...				Turmeric.	stained yellow.	maize ; some	Maize.	masses,
					Granular par-	stained yellow.	Turneric.	some
					ticles of egg			stained.
					protein.			

	Egg Substitute —		Egg Powder.		Egg Substitute—				Rice. 17.	Rice + 1 per Cent. Dried Egg. 18.	Rice + 10 per Cent. Dried Egg. 19.
	10.	11.	12.	13.	14.	15.	16.	18.			
Loss at 100° C. (moisture, etc.)	Per Cent. 10.0	Per Cent. 10.6	Per Cent. 9.97	Per Cent. 11.3	Per Cent. 12.5	Per Cent. 10.08	Per Cent. 11.1	Per Cent. 12.11			
Ash	18.83	11.43	9.3	23.15	20.1	12.06	10.8	0.35			
Calcium sul- phate	0.62	0.24	0.27	1.37	0.091	0.27	0.59	0.35			
CaSO ₄ in ash	3.4	2.1	2.9	5.9	0.45	2.2	5.4	0.35			
Ethereal extract	0.27	1.18	0.80	0.19	0.61	1.15	0.69				
Refraction (Zeiss) at 35° C.											
Nitrogen ...	0.58	0.80	0.90	0.72	0.87	1.13	1.42				
Protein ...	3.62	5.0	5.62	4.5	5.43	7.06	8.87				
Organic phos- phorus (P ₂ O ₅)	0.007	0.017	0.0127			0.0059					
Arsenic (grains per pound) ...	3.00	3.00	1.000	3.30	3.00	1.000	1.000				
Cold water ex- tract	Light yellow	Colourless	Colourless	Yellow	Colourless	Yellow	Deep yellow				
Tartrates ...	+	+	+	-	-	+	-				
Insoluble mat- ter, phosphates	+	-	-	+	+	-	+				
Microscope ...	Wheat and rice masses, Potato stained and unstained. Maize. Turmeric.	Rice masses and grains. Turmeric.	Rice masses and grains. Turmeric.	Rice masses. Maize stained yellow.	Rice masses. Maize. Turmeric.	Maize, some stained. Parenchyma. Spiral vessels and pigment cells.	Wheat. Rice masses. Some wheat stained yellow.		Rice masses and grains.		

egg powder of a few grains of turmeric, or, in some cases, a few grains of coloured maize starch. The examinations of baking powders predominated, but a sufficient number of the egg powders were examined to justify the conclusion that egg powder is merely coloured baking powder.

	Baking Powder.			Egg Powder.		
	1.	2.	3.	1.	2.	3.
Ash, per cent.	28.8	14.1	8.6	26.2	20.1	8.95
Calcium sulphate, per cent.	0.78	0.62	0.35	0.7	0.89	0.30
Calcium anphate as percentage of ash ...	2.7	4.4	4.1	2.7	4.4	3.4

Where, then, is the justification for the use of the word "egg" in this substance? That there was probably some such idea in the public mind suggested itself to some trader, as a sample was recently brought into these laboratories bearing a small covering label with the seductive suggestion that it contained "the actual egg." For the purpose of testing the accuracy of this statement and obtaining information to see if it would be possible to separate this substance from baking powder and really standardise it, a series of analyses of the various brands sold here was made and compared with the analytical results obtained from the examination of "desiccated egg," both purchased and prepared.

The following analytical figures were determined for both "desiccated egg" and egg powders, some of which were described as egg substitute—an equally unsatisfactory name, as a coloured baking powder is no satisfactory substitute for "eggs": Moisture, ash, calcium sulphate, ethereal extract, nitrogen expressed as protein, organic phosphorus, and microscopical character.

The appended table shows the results obtained in the case of three samples of desiccated egg and fourteen samples of egg powder and substitute. A comparison of these figures shows how widely these substances differ in all essential features, there being no evidence of egg whatever in any egg powders except that stated to contain actual egg, and then the evidence is limited to microscopical detection, the increase in ether extract, protein and organic phosphorus being so small as to be negligible. "It is quite clear that in any revision of values this substance would have no place; for a real claim to the use of the word 'egg' in this connection should mean a definite figure for ether extract, protein and organic phosphorus." With regard to the latter figures, in a recent paper in the *J. Amer. Chem. Soc.*, 1920, 42, 2238, Messrs. Brauns and MacLaughlin have shown that lecithin and cephalin can be extracted from organic substances and examined; but as the process suggested would involve too great a labour, the extraction by means of hot alcohol of these substances, their saponification in the alcoholic solution, and the estimation of the total phosphorus would appear adequate, and by this means a standard could be laid down for a minimum percentage of actual egg. In the last column of the table the effect of mixing dried eggs with the usual base of egg powder, the cereal

rice, is studied, and we would suggest that the figures of this last column provide a suitable basis for a standard.

DISCUSSION.

Mr. G. RUDD THOMPSON said that it was a very open question whether so-called "egg powders" were not a very grave infringement of the Food and Drugs Act. Action had already been taken where there was a specific declaration of "egg powder" or "containing the active principle of the egg," but that, as a rule, manufacturers did not give this loophole; they used instead such a phrase as "A perfect substitute for egg." He considered that a thorough report should be made on all these substances put on the market, as so many of them were mere frauds, being composed of nothing more than coloured starch, or baking powder, to which methyl orange had been added.

Dr. DYER said that for a great number of years there was no such thing as egg powder in the literal sense, and during that time (when it could not possibly have been misleading) the term "egg powder" had come to be applied to baking powder coloured yellow. More recently, powdered egg had become possible, which had led to a certain amount of ambiguity between egg powder and powdered egg, and it was difficult to say that it was not of the nature under which it was sold. He did not think the authorities were slack. But there was doubt as to whether the long continuance of the term "egg powder," as applied to coloured baking powder, might not legally be held to have established something in the nature of a commercial "right of user" of this title.

Dr. VÖELCKER alluded to a case he had come across lately of a material which appeared to consist of genuine egg powder, and which, though unsuitable for human use, was sold to farmers as a food for poultry. It was perfectly genuine, consisting of egg powder giving an ether extract of 39 per cent., and albuminoids 43 per cent., was sold at a reasonable price, and was a good food for poultry. He would welcome anything which could be done to establish a firm understanding on this matter.

Mr. MORE said it was well known commercially that an egg powder was not an "egg" powder, but a baking powder coloured with a small quantity of aniline dye. He considered that some powers were required in the new Food Bill to ensure that such materials were properly and correctly named.



NOTES.

The Editor desires to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

A SIMPLE METHOD FOR TESTING GLUE.

MANY methods for testing glue have been suggested, and most of them are unsatisfactory or give results which are difficult to interpret.

The following simple method involving the use of the machine for testing the tensile strength of cement has been devised, and is claimed to be a useful addition to the tests already in use.

The method is as follows: Briquettes of pitch pine of the same dimensions as those used in testing the tensile strength of cement (*cf.* British Standard Specification for Cement) are made, the grain running longways. These are then sawn across the middle at right angles to the axis, care being taken that the operation is done with great exactitude, and the cut surfaces smoothed down with sand-paper so that they fit absolutely when placed together.

Twenty-five grms. of glue are allowed to stand in 100 c.c. of water for twenty-four hours. This is then heated on a water-bath until all the glue has dissolved, the evaporated water replaced, and the solution cooled to 50° C. A quantity of the solution is then removed by a small pipette specially made from a thin glass tube delivering exactly 0.3 c.c., which is found to be the amount required to cover the sq. inch section of the briquette. This amount is run out on to the flat surface of one of the half-briquettes, the other half is put immediately on top of it, and the two halves are held in position by a 2-inch rubber band. After twenty-four hours the briquette is placed in the cement testing machine and broken in the ordinary way.

The cross-section of the middle of the briquette being exactly 1 sq. inch, the tensile strength of the glue can be given in pounds per sq. inch. Several briquettes should be used for each sample, and the mean results taken.

The following are the results obtained from ten samples of glue. The figures marked * are considered abnormal and not included in the means.

1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	
Pounds per Square Inch.										
410	300	60	400	*500	400	40	370	100 ⁵	450	
445	180	0	280	190	250	60	328	120	410	
575	180	115	380	175	265	120	315	105	300	
440	220	75	255	280	305	90	450	140	470	
*275	220	110	430	220	*605		380		450	
550	365	110	446	215	*175					
480	270	50	285	170	400					
440	280	60	380	210	405					
Mean :	477	246	72	356	207	338	78	369	124	416

In order to classify the glues for general purposes, the following standards were made:

Quality.		Tensile Strength. Pounds per Square Inch.	
A. Good	...	More than 300	
B. Medium	...	150 and less than 300	
C. Poor	...	Less than 150	

CONCLUSIONS.—1. From the above results it will be seen that there is an occasional abnormal briquette; obviously this should be omitted from the mean of the series.

2. That by the above method anyone can fix his own standards, using whatever wood or strength of glue be most suitable.

3. It is suggested that for each series six briquettes be made.

4. That a really fair estimate of the strength of different glues can be obtained by this method.

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THE ESTIMATION OF SMALL AMOUNTS OF CHROMIUM IN STEELS.*

Since the publication of my paper on the estimation of small quantities of chromium in steel (ANALYST, 1921, 38), my attention has been drawn to the sensitive colour reaction of chromic acid with diphenyl-semicarbazide described by Cazeneuve (ANALYST, 1900, 25, 331). This indicator gives an intense "permanganate" colour with chromic acid, visible at far greater dilutions than the colour of the chromic acid itself. It was thought worth while to try the chromium process, using this indicator in the colour matching, and to carry it to greater dilutions, to find out if the separation of the chromium from the iron is as complete as it appears to be.

Samples containing a small but measurable quantity of chromium gave identical results, whether the chromium was estimated in the ordinary way by direct matching of the chromic acid colour, allowing for the blank, or whether the indicator was added to the two tubes and the purple colour matched (if necessary diluting and taking an aliquot part).

The details of the experiments, with percentages of chromium too low to be estimated by matching the chromic acid colour, are as follows:

$\frac{N}{1000}$ $K_2Cr_2O_7$ added to 4 Grms.	Cr added.	$\frac{N}{1000}$ $K_2Cr_2O_7$ re- quired for 100 c.c.	$\frac{N}{1000}$ $K_2Cr_2O_7$ re- quired for 4 Grms.	Cr found.
c.c.	Per Cent.	c.c.	c.c.	Per Cent.
Blank	—	Less than 1 drop	—	—
1.00	0.00043	0.15	0.75	0.00032
2.00	0.00087	0.35	1.75	0.00076
3.00	0.00130	0.55	2.75	0.00120
4.00	0.00174	0.70	3.50	0.00152

In each case 4 grms. of electrolytic iron were used, and the separation carried out as in the original experiments.

These results prove that the oxidation and separation of the chromium from the iron are complete.

The indicator is made up by dissolving 1 grm. of diphenyl-semicarbazide in 10 c.c. glacial acetic acid, and making up the solution to 1,000 c.c. with water; 5 c.c. are added to each tube, and 10 c.c. of 1 : 3 sulphuric acid are used instead of the original 20 c.c.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Colorimetric Estimation of Lactose in Milk. R. G. Owen and R. Gregg. (*J. Lab. Clinical Medicine*, 1921, **6**, 220-221; *Chem. Abstracts*, 1921, **15**, 1033.)—The method described is a modification of that employed by Folin for the estimation of sugar in blood (*ANALYST*, 1920, **45**, 227). Two standard solutions of pure lactose are prepared containing 0.25 and 0.35 mgrm. per c.c. To 1 c.c. of milk 2 c.c. of sodium tungstate solution and 2 c.c. of $\frac{2}{3}$ *N* sulphuric acid are added, and the mixture allowed to stand for five minutes, diluted to 100 c.c., and filtered. One c.c. of the filtrate is run into a special sugar tube, 1 c.c. of water being added. Into two similar tubes 2 c.c. of each of the standard solutions are run, and 2 c.c. of alkaline copper solution are added to the three tubes, which are then immersed for six minutes in a boiling water-bath. Two c.c. of Folin's molybdate-phosphate solution are introduced, and the mixtures are diluted to 25 c.c. The colour of the solution under examination is then compared in a colorimeter with the standard to which it most closely approximates.

T. J. W.

"Rice" Beans (*Phaseolus calcaratus*). (*Bull. Imp. Inst.*, 1920, **18**, 476-478.)—*Phaseolus calcaratus* is widely cultivated in India, where it is known as the "rice" bean. Five samples of beans of different varieties gave the following results: Moisture, 9.0 to 10.6; crude proteins, 20.8 to 25.3; fat, 0.5 to 1.0; carbohydrates, 54.7 to 60.3; fibre, 4.8 to 5.4; ash, 3.8 to 4.6 per cent.; nutrient ratio, 1 : 2.9 to 1 : 2.2; food units, 113 to 119. All the samples were free from hydrocyanic acid. These beans are similar in composition to "black gram" (*Phaseolus Mungo*) and cow-peas (*Vigna Catjang*), and would be readily saleable in the United Kingdom as a feeding-stuff.

R. G. P.

Adulteration of Rice Bran. (*U.S. Dept. Agriculture. Service and Regulatory Announcements.* Suppl. N.J. 8,431, March 10, 1921.)—Two shipments of rice bran had the following composition: Moisture, 8.48 and 7.83; ether extract, 12.03 and 13.04; crude fibre, 16.70 and 17.52; crude protein, 10.66 and 10.88; ash, 12.33 and 12.15; and ash insoluble in acid, 7.40 and 7.32 per cent. These analyses indicating the presence of added rice hulls, proceedings were taken, and a conviction obtained.

Hydrocyanic Acid as a Sterilising Agent for Flour. Marchadier and Goujon. (*J. Pharm. Chim.*, 1921, 23, 417-420.)—Whilst recognising the value of gaseous hydrocyanic acid as a disinfectant for fabrics and clothing, the authors contend that it is unsuitable for farinaceous foodstuffs on account of the possible retention of small quantities of the cyanide, and the fact that flour so sterilised does not make good bread. They have examined a sample of wheat flour having a distinct smell of almonds, which persisted even in foods prepared from it. The sample gave a strong Guignard reaction with sodium picrate paper, and showed the presence of 82 mgrms. of hydrogen cyanide per kilo. Such quantity is dangerous to the consumer, and treatment of flour in this way should be prohibited. H. E. C.

Use of Dyes in the Microscopical Examination of Bread, Flour, Feeding Stuffs, etc. P. P. Fechner. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1921, 41, 170-172.)—A dye reagent, termed "Violet," is recommended as being useful for the ready identification of the various constituents of bread, flour, etc.; when treated with this reagent, the cellulose tissue is coloured blue-green to blue-violet, the proteins violet or red, the oil yellow or brown, whilst the starch remains white or colourless. Another reagent, also prepared by the author, colours the cellulose tissue red, and differentiates to some extent between the different varieties of starch; rye, wheat, barley, oat, rice, maize, millet, and buckwheat starches are coloured black or brown, whilst sago, banana, potato, acorn, chestnut and leguminous starches remain white in colour. W. P. S.

Cassava Flour. C. Grimme. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1921, 41, 172-175.)—Bitter cassava (*Manihot utilisima*), when dried, is ground to form cassava flour; further preparation yields starch (tapioca) and alcohol. Cassava flour contains: Water, 12.73; proteins, 0.65; fat, 0.11; starch, 88.84; crude fibre, 1.81; and ash, 1.12 per cent. The ash contains about 33 per cent. of K_2O and 20 per cent. of P_2O_5 . W. P. S.

Estimation of Carbohydrates in Vegetable Foods. V. C. Myers and H. M. Croll. (*J. Biol. Chem.*, 1921, 46, 537-551.)—*Soluble sugars*: The vegetable is finely chopped, well mixed, and ground in a mortar with sand and a measured volume of water. A portion of the mixture is saturated in the cold with pure picric acid and filtered. Two 3 c.c. portions of the filtrate are measured into test-tubes, and 1 c.c. of 22 per cent. sodium carbonate solution is added to one of them, both tubes being then heated in a boiling water-bath for ten minutes. One c.c. of sodium carbonate solution is run into the second tube, and the heating of both tubes continued for a further twenty minutes, when they are cooled and diluted to 10, 15, or 20 c.c. to match approximately a standard prepared by heating for twenty minutes 3 c.c. of 0.02 per cent. pure dextrose solution with 1 c.c. of sodium carbonate solution, cooling the liquid, and diluting it to 10 c.c. The colours are matched in a colorimeter, and any increase shown in the solution hydrolysed by heating with picric acid represents the sucrose present. *Total available carbohydrate*: The ground vegetable is weighed out accurately into a graduated flask, and

boiled with 100 c.c. of water for one and a half hours under a reflux condenser, then ground to a paste, and rinsed back into the flask with water to give a total volume of about 200 c.c. A plug of cotton-wool is inserted in the neck of the flask, and the contents are heated to boiling and cooled to 40° C., after which 5 c.c. of a 1 per cent. solution of taka diastase and a few c.c. of toluene are added, and after its contents have been mixed the flask is kept at 37° C. for eighteen hours. The mixture is then cooled, diluted to 250 or 300 c.c., and filtered after thorough shaking, after which 20 c.c. of the filtrate are heated with 1.4 c.c. of concentrated hydrochloric acid in a boiling water-bath for one or two hours. After cooling, the liquid is almost neutralised with strong sodium hydroxide solution, diluted to 25 c.c., and saturated with picric acid, the subsequent procedure being as described above for soluble sugars. A correction must be made by means of a blank estimation for the sugar produced by hydrolysis of the taka diastase solution. Results obtained by this method with a variety of fruits and vegetables are given.

T. J. W.

Inversion and Estimation of Cane Sugar. A. R. Rose. (*J. Biol. Chem.*, 1921, 46, 529-535).—The solution containing less than 5 per cent. of cane sugar is mixed with twice its volume of saturated picric acid solution, and heated in a boiling water-bath for ten minutes. After cooling, the solution may be diluted to a definite volume and a polarimetric reading taken, or a volume of 20 per cent. sodium carbonate solution equal to that of the sugar solution taken may be run in, the mixture heated in the water-bath for thirty minutes, and the red colour produced compared in a colorimeter with that given by a standard solution of pure sugar (Dehn and Hartman, *J. Amer. Chem. Soc.*, 1914, 36, 403). Should dextrose or lævulose be present in the original solution, the cane sugar may be estimated by polarimetric readings taken before and after the picric acid inversion. For the estimation of sugars in food products, from 1 to 10 grms. of the sample are triturated in a mortar with 100 c.c. of water and 0.5 gm. of solid picric acid. The mixture is centrifuged or filtered, and 1 c.c. of the clear solution is transferred to a test-tube containing 2 c.c. of saturated picric acid and 1 c.c. of 20 per cent. sodium carbonate solution, and heated in a boiling water-bath for thirty minutes, when the red colour produced is proportional to the amounts of dextrose and lævulose present. A similar volume of the solution is hydrolysed by heating for ten minutes with the picric acid before the addition of the sodium carbonate, when the colour produced by reduction will be proportional to the original dextrose and lævulose, together with the invert sugar derived from the sucrose present. Dextrin and maltose, if present, should be removed or allowed for. The results given show that the method yields a high degree of accuracy.

T. J. W.

Grape-Seed Oils. É. André. (*Comptes rend.*, 1921, 172, 1296-1298).—Eleven samples of grape-seed oil, obtained in some cases by pressing the seeds and in others by extraction with petroleum spirit, have been examined. The limiting values found for the various constants are as follows: Sp. gr. at 20°/0° C., 0.9170 to 0.9334; n_D^{20} , 1.4708 to 1.4772; saponification value, 171.0 to 191.1; iodine value, 94.3 to 135.0; saponification value of the acetylated oil, 189.7 to 231.8; acetyl value, 13.3 to 49.3.

T. H. P.

Perilla Seed from Cyprus. (*Bull. Imp. Inst.*, 1920, 18, 479-481.)—Perilla seed (*Perilla ocymoides*), grown experimentally in Cyprus from seed of Japanese origin, contained 6.3 per cent. of moisture and 43.1 per cent. of oil. The oil had the following values: Sp. gr. at 15°/15°, 0.9298; $[n]_D^{20}$, 1.472; saponification value, 190.5; and iodine value, 185. The sp. gr. is lower than that previously recorded for perilla oil—viz., 0.9318 to 0.9372; otherwise the figures closely resemble those recorded previously. The residual meal gave the following results calculated on a basis of 7.0 per cent. of fat: Moisture, 9.0; crude proteins, 35.6; fat, 7.0; carbohydrates, 19.4; fibre, 18.9; ash, 10.1 per cent.; nutrient ratio, 1:1.0; food units, 126. The meal therefore contains rather less fat and considerably more protein than undecorticated cotton-seed cake, and, if found to be non-injurious to animals, would form a valuable feeding-stuff.

R. G. P.

Oil of "Maroola" Nuts (*Sclerocarya Caffra*). (*Bull. Imp. Inst.*, 1920, 18, 481-483.)—The kernels of *Sclerocarya Caffra* (N.O. *Anacardiaceae*), a small tree found only in Natal and the Transvaal, contained 5 per cent. of moisture and 56.2 per cent. of a clear, pale, yellowish-brown oil with the following values: Sp. gr. at 15°/15°, 0.9167; $[n]_D^{20}$, 1.460; m.-pt. of fatty acids, 25.0° C.; acid value, 3.7; saponification value, 193.5; iodine value, 76.6; soluble volatile acids equivalent to 0.1 c.c. $\frac{N}{10}$ alkali; insoluble volatile acids, 0.45 c.c. $\frac{N}{10}$ alkali per 5 grms.; and unsaponifiable matter, 0.6 per cent. As the nuts are difficult to crack, and only contain 10 per cent. by weight of kernel, they are unlikely to be of commercial value as a source of oil.

R. G. P.

Analyses of Chicory and Other Coffee Substitutes. J. Pritzker and R. Jungkunz. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1921, 41, 145-169.)—Analyses are recorded of some fifty-six samples of chicory, roasted beans and cereals, fig coffee, dandelion coffee, etc. In estimating the amount of chicory in a coffee-chicory mixture, use is made of the water-soluble extract of the substances: 10 grms. of the sample are mixed with 200 c.c. of water, weighed, and boiled for five minutes. After cooling, the original weight of the mixture is restored by the addition of water, the solution is filtered, and the specific gravity of the decoction is determined. The percentage of water-soluble substance (TS) is calculated by the formula $TS = (S - 1) \times 5160$, where S is the sp. gr. of the 5 per cent. decoction. The average amount of water-soluble substances yielded by thirty-nine samples of chicory was 60.68 per cent.; coffee gave from 23.2 to 25.2 per cent. Since $\frac{N}{10}$ iodine solution has practically the same colour as that of a 5 per cent. decoction of normal chicory, the author suggests the use of this iodine solution as a standard in comparing the colour of chicory extracts, the colorations being made after suitable dilution. Two samples of dried dandelion root yielded the following results: Water, 10.02, 3.85; ash, 2.02, 5.13; water-soluble substances, 50.00, 36.20 per cent.; and sp. gr. of the 5 per cent. decoction, 1.0097, 1.0070.

W. P. S.

Volatile Oil from the Leaves of the "Wild Pimento" of Jamaica. O. D. Roberts. (*J. Soc. Chem. Ind.*, 1921, 40, 1-96r.)—From the distillation of the

Leaves of *Anemum* (= *Pimenta*) *jamaicensis* about 0.49 per cent. of volatile oil of pale yellow colour, having an odour resembling lavender, was obtained, which had the following constants: Sp. gr. at 15°/15° C., 0.8895; $[\alpha]_D^{25} = -6^\circ 0'$; $n_D^{25} = 1.471$; acid value, 2.4; ester value before acetylation, 4.2; and ester value after acetylation, 129.4. The oil is soluble in 2.5 volumes of 70 per cent. alcohol, and becomes cloudy again with six volumes. The composition of the oil was: Terpenes, including α -phellandrene and dipentene, 17.0; cineol, 15.0; aldehydes, 0.1; alcohols (*L*-linalol and geraniol), 38.3; esters as linalyl acetate, 1.5; phenols, 0.1; acids (acetic and caproic), 0.2; and residue containing sesquiterpenes, 27.8 per cent. H. E. C.

Essential Oil from *Perilla Nankinensis*. S. Furukawa and Z. Tomizawa. (*J. Chem. Ind. Tokyo*, 1920, 23, 342; *J. Pharm. Chim.*, 1921, 23, 426.)—The oil distilled from the fresh plants in different stages of growth shows a gradual increase in content of aldehydes and diminution of esters by the action of an enzyme, whilst the oil obtained from the dried plants changes in the converse way. The oil has the following percentage composition: *L*-limonene, 20 to 30; perillaldehyde, 44 to 57; and a trace of α -pinene. Perillaldehyde has b.-pt., 237°; sp. gr. at 15°/15° C., 0.9675; $n_D^{25} = -145.8^*$, yields an exceedingly sweet α -antialdoxime (m.-pt. 102°) and a β -synaldoxime (m.-pt. 129°) which is not sweet. It also gives a crystalline phenylhydrazone and semicarbazone. Perillonitrile ($C_9H_{13}CN$) is a liquid, as sweet as saccharin, of b.-pt. 123° (15 mm.) and sp. gr. at 15° C. 0.9488 to 0.9490; and perilla alcohol is described as of b.-pt. 118° to 121° C. (11 mm.), and sp. gr. 0.9690. H. E. C.

Bromometric Estimation of Salicylic Acid. I. M. Kolthoff. (*Pharm. Weekblad*, 1921, 58, 699-702.)—In an investigation of the bromometric method for estimating salicylic acid, 25 c.c. of $\frac{N}{10}$ potassium bromate were mixed with 1 grm. of sodium bromide and 25 c.c. of the salicylic acid solution, and varying amounts of 4 *N*-hydrochloric acid were added. After standing for five minutes in a stoppered flask 25 c.c. of *N*-potassium iodide were introduced into the mixture, and it was then titrated with a thiosulphate solution. The results indicated that the accuracy was largely dependent on the amount of hydrochloric acid added. Between 6.5 and 15 c.c. the error was -1.3 to -14 per cent.; dilution and more addition of potassium iodide decreased the error, but accurate results were only obtained with 2 to 6 c.c. of acid. Only a slight excess of bromate is essential; 27 per cent. was found to be sufficient.

Fresenius and Grunhut's method (*Zetsch. anal. Chem.*, 1899, 38, 298) of liberating bromine before adding the salicylic acid is not to be recommended, as too much bromine becomes volatilised. In the estimation of very small amounts of salicylic acid—e.g., in foodstuffs—the amount of hydrochloric acid is of less importance than with stronger solutions; thus, with 10 c.c. 4 *N*-HCl added to 25 c.c. of a 0.001 mol. solution the error was only -2.3 to -2.7 per cent. The following method gives accurate results: Twenty-five c.c. of $\frac{N}{10}$ potassium bromate, 1 grm. of sodium bromide, and not more than 5 c.c. of hydrochloric acid, are added to 25 c.c. of salicylic acid in

* Temperatures not given.

0.012 to 0.008 mol. solution. After the mixture has stood for five to ten minutes in a stoppered flask, 5 c.c. of *N*-potassium iodide are rapidly added, and the liquid is titrated with $\frac{N}{10}$ thiosulphate, starch being added only towards the end of the titration. The addition of alcohol, as in the bromometric estimation of phenol, may be omitted.

W. J. W.

Necessity for the Physiological Control of Adrenaline and Suprarenal Preparations. M. Tiffeneau. (*J. Pharm. Chim.*, 1921, [vii.], 23, 313-317, 366-375.)—Six samples of adrenaline of various origins have been examined, with the following results: (1) Synthetic origin, $[\alpha]_D^{20} = -52^\circ 30'$, physiological activity 100; (2) natural, $[\alpha]_D^{20} = 53^\circ 36'$, physiological activity 100; (3) unknown origin, $[\alpha]_D^{20} = -38^\circ 33'$, physiological activity 60; (4) synthetic, $[\alpha]_D^{20}$ 0, physiological activity 51; (5) synthetic, $[\alpha]_D^{20}$ 0, physiological activity 46; (6) unknown origin, $[\alpha]_D^{20} = -24^\circ 17'$, physiological activity 42. Thus, for *lævo*-rotatory adrenelines, the results of the physiological test correspond well with the optical rotations, whilst for the racemic compounds this is no longer the case. Suprarenal preparations lose in physiological activity if kept under unsuitable conditions, and should be tested before use.

T. H. P.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Lathyrism or Poisoning caused by Vetch Seeds. M. Mirande. (*Comptes rend.*, 1921, 172, 1142-1143. Cf. Hughes, *ANALYST*, 1895, 20, 169.)—The author has investigated the cause of lathyrism, the poisoning produced in man and animals by ingestion of certain vetch seeds, notably those of *Lathyrus sativus* and *L. cicera*. The cotyledons are found to contain a substance which readily undergoes auto-fermentation, with liberation of carbon dioxide and hydrogen sulphide in considerable proportions. Water removes this substance from the crushed seeds, but careful washing is necessary to render the extraction complete. Incipient germination makes the seeds incapable of yielding hydrogen sulphide when subsequently pounded and moistened. The fermentation of the substance is scarcely appreciable at 35°C ., but is very active between 35° and 40°C ., and ceases only at temperatures above about 60°C ., which is the average coagulating temperature of the proteins.

T. H. P.

Occurrence of Catalase in Fish. P. C. Stockstad. (*Tids. Kem.*, 1920, 17, 181-187; *Chem. Abstracts*, 1921, 15, 691.)—Ten species of fish were found to give positive reactions for catalase, those containing the highest content of fat and esterase yielding the highest catalase constant. Tests were made with P_H 7.07 and at a temperature of 15°C ., and the constants determined by titration with potassium permanganate before and after enzymic digestion. The values obtained ranged between 1.19 and 4.8, and in all cases the reaction was monomolecular, but with herring the results were abnormal, owing to the effect of some undetermined factor.

T. J. W.

* Temperature not given.

Oxidising Enzymes of Fruits. M. W. Onslow. (*Biochem. J.*, 1921, 15, 113-117.)—This work is a continuation of that described previously by the author (*Biochem. J.*, 1920, 14, 541-547). The fresh tissues, neutralised aqueous extracts, residues and filtrates from cold alcoholic extracts, and hot alcoholic extracts were purified by precipitation with lead acetate, decomposition of the precipitate with dilute sulphuric acid, and removal of organic acids and tannins by treatment with alcoholic calcium chloride and gelatin, and tested with benzidine, α -naphthol, and guaiacum, each with the addition of hydrogen peroxide, and with catechol and guaiacum. The following fruits were shown to contain the components of an oxidase: Cherry, peach, apricot, medlar, strawberry, grape, fig, and mulberry; whilst a peroxidase only was detected in blackberry, red currant, black currant, gooseberry, grape fruit, pomegranate, pineapple, melon, and tomato. Although the fruits contain no oxidase, this class of enzyme is present in blackberry and tomato leaves. Details are given of the reactions obtained with the tissues and the various extracts obtained from the fruits. T. J. W.

Antiscorbutic Capacity of Milk Powders. E. B. Hart, H. Steenbock, and N. R. Ellis. (*J. Biol. Chem.*, 1921, 46, 309-318.)—Experimental work on the feeding of scorbutic guinea-pigs upon a basal diet to which had been added dried milk prepared by different methods has shown that the original antiscorbutic property of the milk is destroyed by certain methods. The diet was composed of heated alfalfa flour, rolled oats, casein, and salt, the casein being replaced by the milk powder under examination to the extent of 40 per cent. of the mixture. The methods giving a product without antiscorbutic properties were the Merrell-Soule spray process and the uncondensed spray process employed by the California Central Creameries. The Just process, in which the milk is dried on heated rolls, yielded a powder possessing excellent antiscorbutic properties, whether prepared from summer milk or winter milk obtained from cows rationed with roots and tubers. T. J. W.

"Black Spot" of Chilled and Frozen Meat. F. T. Brooks and M. N. Kidd. (*Dept. Scientific and Ind. Research, Food Investigation Board. Special Report*, No. 6, 1921, 6 pp.)—The "black spot" fungus has been found on New Zealand mutton and lamb, Australian mutton and rabbits, and upon beef from the Argentine and South Africa. When isolated and grown in pure culture, it proved to be one of the many forms of the mould *Cladosporium herbarum*, which is common on dead vegetable matter. The spores, which can develop at 22° F. (–5.5° C.), are either oval in form, 8 to 16 $\mu \times 4$ to 5 μ , or roughly spherical, 3.5 to 5 μ in diam., budded off in chains from the former. On meat the fungus develops particularly upon the subcutaneous connective tissue, sometimes penetrating to a maximum depth of 4 mm. It produces no toxic substances during growth, whether upon meat or other substrata, and its presence alone does not render the meat unfit for human consumption. It may, however, be accompanied by putrefactive bacteria, and in that case the meat will be soft and have an unpleasant odour. White moulds (species of *Sporotrichium* and *Oospora*) can also develop on meat at temperatures below freezing-point during prolonged storage.

Germicidal Properties of Milk. (*J. Amer. Med. Assoc.; Amer. J. Pharm.*, 1921, 93, 281-282.)—Fresh raw milk allowed to stand for a short time shows an apparent decrease in the number of bacteria present, but this is not noticed if the milk be heated. Recent investigation by W. H. Chambers has confirmed these observations, and shown that this action varies with milk drawn from different cows, and also depends upon the bacterial species present. When the total number only, and not the species, of bacteria present is considered, a decrease in number may be evident, or an unaffected species may increase rapidly and conceal the germicidal action upon another less numerous species present. Lactic acid organisms are apparently unaffected, whilst other bacteria are restrained, and in some cases decreased in numbers. The germicidal property is destroyed by heating milk for two minutes to 80° to 90° C.

T. J. W.

Estimation of Tryptophane in Blood-Serum and Milk. O. Fürth and E. Nobel. (*Biochem. Zeit.*, 1920, 109, 103-123; *Chem. Abstracts*, 1921, 15, 377.)—A freshly prepared solution containing 0.1 per cent. of tryptophane and 2 per cent. of sodium fluoride is employed. To 2 c.c. of the standard in a graduated cylinder 1 drop of 2.5 per cent. formaldehyde is added, followed by 15 c.c. of concentrated hydrochloric acid and 10 drops of 0.5 per cent. sodium nitrite, the total volume being diluted to 20 c.c. by the further addition of hydrochloric acid, and the solution well mixed. The solution to be estimated is similarly treated, 2 c.c. portions being used, and, after standing from five to ten minutes, the colour is compared with that of the standard in a colorimeter. Any precipitate formed due to the presence of excess of inorganic salts is removed by filtration. By the above method it was shown that globulin contains almost three times as much tryptophane as does albumin, the percentage contained in casein being 2.02, in cow's milk whey from 1.8 to 2.3, and in the proteins of human milk from 3.4 to 6.8.

T. J. W.

Estimation of Sugar in Blood. E. Ponder and L. Howie. (*Biochem. J.*, 1921, 15, 171-174.)—The blood is obtained from the finger, drawn into a 0.2 c.c. pipette, and run into 1 c.c. of water, after which the pipette is filled twice with water and the washings added to the diluted blood. To the mixture 0.2 c.c. of Folin and Wu's sodium tungstate solution (*ANALYST*, 1920, 45, 227) and 0.2 c.c. of $\frac{2}{3}$ N-sulphuric acid are added, the tube being allowed to stand for five minutes, with occasional shaking. The mixture is transferred to a micro-filter, and 0.4 c.c. of the filtrate is run into a narrow-necked tube of about 1.5 c.c. capacity. A similar tube receives 0.4 c.c. of 0.01 per cent. dextrose solution, and 0.4 c.c. of alkaline copper tartrate solution is added to each. The narrow necks of the tubes are sealed off in the blowpipe flame, and the tubes are immersed in a boiling water-bath for six minutes, cooled to 80° C., the necks cut off, and 0.4 c.c. of the phenol reagent (*J. Biol. Chem.*, 1919, 38, 106) is added. After cooling, the contents of the tubes are each added to 3.8 c.c. of water, and the colour of the solutions matched in a colorimeter. The results obtained are in excellent agreement with those given by the original Folin and Wu method (1920), and the advantages claimed for the method are the small amount of blood required, the accuracy with which the volume of blood used is known,

the prevention of reoxidation during boiling, and the similarity in colour finally obtained between the standard and the blood solution. T. J. W.

Estimation of Sodium in Small Volumes of Serum. B. Kramer and F. F. Tisdall. (*J. Biol. Chem.*, 1921, 46, 467-473.)—The reagent employed is prepared by adding 10 grms. of potassium pyroantimonate to 500 c.c. of boiling water, boiling the liquid for a few minutes, and, after cooling, adding 15 c.c. of 10 per cent. potassium hydroxide. The filtered solution is kept in a bottle treated with paraffin wax, and will remain in good condition for at least one month at the ordinary temperature. Eleven mgrms. of sodium are precipitated by 10 c.c. of the reagent. To 2 c.c. of serum in a platinum dish 10 c.c. of the potassium pyroantimonate solution are added, followed by 3 c.c. of 95 per cent. alcohol, which is run in slowly with continuous stirring. After standing forty-five minutes, the precipitate is transferred to a Gooch crucible packed with alternate layers of filter-paper and asbestos, and which has been previously used for several similar precipitations, the last portions of the precipitate being washed in with a few c.c. of 80 per cent. alcohol. After drying for one hour at 110° C., the crucible is cooled and weighed. Although calcium phosphate is precipitated from the serum by the alkaline reagent, the amount present is so small that the error introduced is negligible. Estimations may be made with a maximum error of ± 2.5 per cent. of the actual amount of sodium present; and the same results are obtained whether the original serum or its ash be used. T. J. W.

Estimation of Inorganic Phosphate in Urine by Alkalimetric Titration. C. H. Fiske. (*J. Biol. Chem.*, 1921, 46, 285-295.)—A volume of urine containing from 2 to 7 mgrms. of inorganic phosphorus is diluted with water to about 10 c.c. and treated with 1 c.c. of magnesium citrate reagent and 2 c.c. of concentrated ammonium hydroxide, the tube being gently shaken for fifteen minutes. The mixture is filtered under reduced pressure through a Soxhlet filter-tube containing a thin pad of paper pulp, and the precipitate in the tube and on the filter is washed with 10 c.c. of 2.5 per cent. ammonia solution and four 5 c.c. portions of 95 per cent. neutral alcohol. The precipitate is dissolved in $\frac{N}{10}$ hydrochloric acid added in 1 c.c. portions, and the pulp filter added to the solution, being washed in with 2 c.c. of methyl red solution and 13 c.c. of water. The addition of $\frac{N}{10}$ hydrochloric acid is continued until the solution remains acid in reaction after thorough shaking, and the whole solution is titrated with $\frac{N}{10}$ sodium hydroxide until the colour is slightly more yellow than a standard consisting of sodium acetate solution containing a slight excess of acetic acid and methyl red. Each c.c. of $\frac{N}{10}$ hydrochloric acid neutralised by the precipitate is equivalent to 1.552 mgrms. of phosphorus in the sample. The magnesium citrate reagent is prepared by dissolving 4 grms. of magnesium oxide in 100 c.c. of hot water containing 80 grms. of citric acid, cooling the solution, adding 100 c.c. of concentrated ammonium hydroxide, and diluting the mixture to 300 c.c. The methyl red solution contains 0.004 per cent. of the indicator in 50 per cent. alcohol. Experiments made with solutions containing known amounts of potassium dihydrogen phosphate and

with urines in comparison with the von Lorenz method (*Zeitsch. anal. Chem.*, 1907, 46, 193), have shown that accurate results are obtainable.

T. J. W.

AGRICULTURAL ANALYSIS.

Estimation of Citrate-Soluble Phosphoric Acid in Superphosphates. Official Method of the German Agricultural Experiment Station. (*Chem. Zeit.*, 1921, 45, 487.)—The citrate solution is prepared by dissolving 173 grms. of crystallised citric acid in water, adding 536.9 c.c. of ammonia (sp. gr. 0.96), and diluting the solution to 1 litre. Each 2.5 c.c. of this solution must contain 0.1050 gm. of nitrogen. A weighed quantity of 2.5 grms. of the superphosphate is ground in a basin with 25 c.c. of water, the solution decanted through a filter into a 250 c.c. flask, and the insoluble portion treated twice in a similar way. The insoluble portion is then rinsed on to the filter and washed until the filtrate measures about 200 c.c.; if the filtrate is turbid, a drop of nitric acid is added and the solution then diluted to 250 c.c. The filter and insoluble portion are transferred to a flask, 100 c.c. of the citrate solution are added, and the mixture is shaken until the filter-paper is disintegrated; after standing for fifteen hours at the ordinary temperature, the mixture is heated for one hour at 40° C., cooled, diluted to 250 c.c., and filtered. Fifty c.c. of this solution and 50 c.c. of the aqueous extract obtained previously are mixed, and the phosphoric acid estimated by the molybdate method or the citrate method. The reagent used in the citrate method contains magnesium chloride 55.0 grms., ammonium chloride 70.0 grms., citric acid 61.6 grms., and ammonia (sp. gr. 0.91) 625 c.c. per litre; 25 c.c. of this reagent are required for the precipitation, and the ammonium magnesium phosphate is collected after eighteen hours.

W. P. S.

Rapid Combustion Method for the Estimation of Organic Matter and Organic Carbon in Soil. J. W. Read. (*J. Ind. Eng. Chem.*, 1921, 13, 305-307.)—One gm. of soil is digested for five minutes with 6 portions of dilute hydrochloric and hydrofluoric acid solution, the supernatant liquid being poured through a perforated platinum combustion boat containing a pad of asbestos. The residue is completely transferred to the boat and dried at 100° C. for sixteen hours, cooled in a phosphorus pentoxide desiccator, and weighed. It is then transferred to a silica combustion tube containing contiguous columns of cerium oxide asbestos and pumice, copper oxide, cerium oxide asbestos, and a roll of fine copper gauze filled with lead peroxide. The air-purifying train is filled with calcium chloride, soda-lime, and phosphorus pentoxide, whilst the absorption apparatus contains sulphuric acid pumice, phosphorus pentoxide, soda-lime, calcium chloride, and palladium chloride solution. The combustion is carried out in a rapid current of air and occupies no more than twenty minutes, the total time, including the initial and final weighings, not exceeding forty minutes. By determinations upon pure sucrose it was shown that the results obtained were of a high order of accuracy.

T. J. W.

ORGANIC ANALYSIS.

Estimation of the Organic Carbon in Bituminous Limestones. E. Sernagliotto. (*Giorn. Chim. Ind. Appl.*, 1921, 3, 153-154.)—The following modification of the Corleis method for determining the total carbon in steel gives good results when applied to the estimation of the organic carbon in bituminous limestones. The Corleis flask, coated externally with asbestos and furnished with an internal condenser, is connected on one side with a tower charged with lump potash and on the other, in succession, with a sulphuric acid drying tube, a short, hard glass tube containing cupric oxide and lead chromate, a phosphoric anhydride tube, a soda lime tube, and a phosphoric anhydride tube. A weighed quantity of the material containing about 0.1 gm. of organic carbon is introduced into the flask by means of a long, wide-necked funnel, which is rinsed down with 30 c.c. of syrupy phosphoric acid, and then with water from a wash-bottle; the flask is then closed by a rubber stopper traversed by two glass tubes. The carbon dioxide liberated is eliminated by warming the flask and repeatedly evacuating it with a water-pump, and is replaced each time by air which has passed through the potash tower. The condenser is then fitted, the sulphuric acid seal in the neck of the flask re-established, and connection made with the absorption apparatus, the copper oxide tube being heated to redness. Meanwhile pure chromic acid is dissolved in an equal weight of water, and 35 c.c. of the solution thus obtained boiled for half an hour with 200 c.c. of pure concentrated sulphuric acid (sp. gr. 1.84). When cooled, the liquid is introduced into the Corleis flask and heated gently to boiling, which is maintained for about four hours, the subsequent operations being as in the estimation of carbon in cast-iron.

T. H. P.

Separation and Detection of Lactic Acid as Sodium Ferric Lactate. K. A. Hoffman. (*Ber.*, 1920, 53, 2224-2226; *Chem. Abstracts*, 1921, 15, 813.)—The solution under examination is treated with ferric chloride in slight excess, and the mixture rendered alkaline with sodium carbonate. The solution is then acidified with acetic acid, concentrated on a water-bath, and allowed to stand several hours, when a pale green precipitate of sodium ferric lactate will be formed, even when only small amounts of lactic acid are present. The precipitate is practically insoluble in water, is unaffected by 15 per cent. acetic acid, but is attacked slowly by dilute ammonia solution. It is readily decomposed by stronger alkalis and acids.

T. J. W.

Detection of Oxalic, Tartaric, and Formic Acids in Presence of Each Other. F. Kräuss and H. Tampke. (*Chem. Zeit.*, 1921, 45, 521.)—The detection of oxalic acid by the violet coloration produced with resorcinol and concentrated sulphuric acid (Brauer, *ANALYST*, 1920, 45, 338; Chernoff, *ANALYST*, 1920, 45, 422) is affected by the presence of nitrites and nitrates and of iron salts; the former should first be reduced with nascent hydrogen, and the latter rendered inactive by boiling with sulphurous acid. Tartaric acid gives a deep red coloration with a 1 per cent. solution of resorcinol in sulphuric acid; even if iodides, nitrites,

and nitrates be present, a red ring may be obtained by carefully heating the mixture at the zone of contact of the reagents. Similarly, an orange-coloured ring is formed with formic acid. For the detection of oxalic, tartaric, and formic acid in a possible mixture of these, carbonates, sulphur, iodine, bromine, and other oxidising agents must first be removed, and 0.2 gram of pure resorcinol is then added to a solution of the substance in 5 c.c. of dilute sulphuric acid, after which 10 c.c. of concentrated sulphuric acid are introduced. If all three acids be present, formic acid will be indicated by an orange ring which gradually expands. A narrower blue ring below this will denote oxalic acid, whilst careful heating of the sulphuric acid will give rise to the formation of a deep red ring, due to tartaric acid. W. J. W.

Analysis of Partly Hydrolysed Fats. W. Fahrion. (*Chem. Umschau Fette Oele*, 1921, 28, 68-69.)—The errors which may occur in the calculation of the results of analysis of mixtures of neutral fat and free fatty acids such as are produced by fat-splitting or in so-called "acid oil" (*i.e.*, the mixture resulting from the acidification of soap stock obtained by the removal from oils of free fatty acids by means of alkali) are discussed in detail. If the free fatty acid content of the mixture be calculated from the ratio of the acid value to the saponification value, an error is introduced, because the equivalent weight of fatty acid is less than that of its glyceride by the equivalent weight of the glyceryl radicle, whilst another source of error is the small difference between the neutralisation and saponification values of free fatty acids; in many cases the errors compensate one another, and the results are sufficiently accurate for technical purposes. The following method is suggested: The acid value is determined in the usual way, alcoholic alkali is next added, and the saponification value is then determined; finally, the liquid is rendered alkaline, diluted to an alcoholic content of 50 to 20 per cent., and unsaponifiable matter (x) is estimated by extraction with petroleum spirit or ether. The percentage of free fatty acid (x) is then calculated from $x = (100 - y) \times \text{acid value} / \text{saponification value}$, and the neutral fat from $100 - (x + y)$. R. G. P.

INORGANIC ANALYSIS.

Colorimetric Method for the Estimation of Hydrogen-Ion Concentration in Small Volumes of Liquid. L. D. Felton. (*J. Biol. Chem.*, 1921, 46, 299-305.)—Drops of the liquid under examination are placed upon a glazed porcelain tile, and 1 drop of indicator solution is allowed to fall vertically upon it, complete mixture being attained by means of a small glass rod. After noting the colour produced, drops of various buffer solutions judged to give the same colour are placed close to the original drop, and the colours compared after the addition of a drop of indicator solution to each. The indicators are prepared by mixing equal volumes of 0.02 or 0.04 per cent. solutions of the dyes in 25 per cent. alcohol, and the following mixtures were employed: Thymol blue and bromphenol blue, giving a range of P_H from 1.2 to 4.6; methyl red and bromthymol blue, 4.6 to 7.6; methyl red and bromcresol purple, 4.6 to 7.0; and methyl red and thymol blue, 4.6 to 9.0. Tests made by the method show that an accuracy of at least 0.1 P_H could be obtained. T. J. W.

Reduction of Arsenic Halogen Compounds. O. Ruff. (*Chem. Zeit.*, 1921, 45, 522-523.)—If arsenic trichloride be heated with aluminium chloride and yellow phosphorus to 150° C., a red compound, containing arsenic, aluminium, chlorine, and phosphorus, is formed, from which the phosphorus can readily be separated. If the phosphorus be replaced by metallic aluminium, a similar compound having the formula $AlAs_2Cl_3$ is produced, from which the aluminium chloride may be removed by means of solvents. In each case finely divided arsenic is simultaneously obtained.
W. J. W.

Colorimetric Estimation of Bismuth in High-Grade Ores. G. Spurge. (*Chem. Age*, 1921, 4, 584.)—The colorimetric method, based on the yellow colour produced by the addition of potassium iodide to a dilute solution of bismuth, and originally devised for the estimation of traces of bismuth, has been found suitable for the direct estimation of bismuth in high-grade ores. The ore is finely pulverised in an agate mortar, and 0.05 grm. in the case of ores containing over 3 per cent. of bismuth, or 0.50 grm. for those under 3 per cent., is dissolved by boiling with 25 c.c. of nitric acid and 75 c.c. of water for ten minutes, and, after cooling, the solution is made up to 500 c.c. and filtered. The filtrate is then run from a burette into a Nessler tube containing 10 c.c. of 10 per cent. potassium iodide solution and 3 drops of sulphurous acid until the colour matches that obtained under similar conditions in another tube containing 1 c.c. of standard bismuth solution, the liquid in both tubes being diluted to 50 c.c. before the final matching of colour. The standard solution is made by dissolving 0.025 grm. of pure bismuth as described above, and diluting the solution to 500 c.c. (1 c.c. = 0.00005 grm. bismuth). An estimation can be made in about fifteen minutes, and accurate results are easily obtained with ores containing up to about 20 per cent. of bismuth, though considerable practice is necessary for high-grade ores. The following are typical results, showing how the method compares with the oxychloride method: (1) 45.36; (2) 31.92; (3) 43.80; (4) 13.10; (5) 14.00; (6) 16.60; colorimetric method: (1) 45.5; (2) 31.8; (3) 43.5; (4) 12.8; (5) 13.8; (6) 16.3 per cent. Copper in large quantities may give a precipitate, which can be filtered off; this is generally unnecessary, as the solution is too dilute and the titration too rapid for the separation of any copper iodide. Lead, if present in quantity, might interfere with the estimation, but could, if necessary, be removed as sulphate.
R. G. P.

Electrolytic Estimation of Gold and its Separation from Copper, Palladium, and Platinum. W. D. Treadwell. (*Helv. Chim. Acta*, 1921, 4, 364-374.)—Gold may be deposited electrolytically from a faintly acid chloride solution to which 1 to 2 c.c. of strong hydrochloric acid and 1 to 2 grms. of ammonium acetate have been added; a platinum wire gauze cathode and platinum spiral are used. The operation is begun in the cold with 1 to 1.3 volts and 0.1 to 0.15 amp.; the temperature is gradually raised to 50° to 60° C. Deposition is complete in twenty minutes if nitrogen or carbon dioxide be bubbled through the electrolyte; the current density drops to 0.001 amp. when all the gold is precipitated. This procedure provides a quick and accurate separation from copper. For the separation from

palladium the chloride solution, prepared as above, is electrolysed with 0.4 to 0.5 volt at 50° to 55° C. After deposition of the gold, the electrolysis is continued for palladium with a fresh platinum cathode and a pressure of 1.2 volts. Gold is separated from platinum in a chloride bath by substituting sodium acetate for the ammonium salt, using a platinum dish and a rotating platinum disc anode, and electrolysing at 0.7 volt pressure while warming to 50° to 60° C. For the subsequent determination of platinum, the solution is transferred to a clean platinum dish and electrolysed with a current of 1.5 volts. Gold may be separated in this manner from palladium and platinum.

W. R. S.

Volumetric Estimation of Zinc. I. M. Kolthoff and J. C. van Dijk. (*Pharm. Weekblad*, 1921, **58**, 538-553.)—In a direct acidimetric estimation, the best results (accurate to 1 per cent.) were obtained by adding 25 c.c. of $\frac{N}{10}$ zinc sulphate to 30 c.c. of $\frac{N}{10}$ sodium hydroxide, boiling for three minutes, and titrating with $\frac{N}{10}$ hydrochloric acid, with phenolphthalein as indicator. The indirect estimation of zinc, by precipitation with hydrogen sulphide and titration of the free acid in presence of an iron salt, gives fairly accurate results if the titration be carried out with sodium borate with brisk agitation. If an indicator be substituted for the ferrous salt, only iodoeosin is suitable. The estimation of zinc as mercury zinc thiocyanate gives the best results, provided chlorides be absent. With concentrations above $\frac{N}{100}$, the solution may be filtered immediately after adding the reagent, the excess being titrated with mercuric nitrate. If the zinc solution be weaker than $\frac{N}{100}$, the filtrate must be allowed to stand for one day, and the reagent, which must have a concentration of at least 0.006 N to render the solubility of the double salt negligible, is then titrated back by Volhard's method. The presence of sulphuric or nitric acid is not detrimental if the solution be filtered immediately.

W. J. W.

Estimation of Zinc by the Ferrocyanide Method. E. Olivier. (*J. Soc. Chem. Ind.*, 1921, **40**, 107-108t.)—The zinc in Australian concentrates is generally estimated volumetrically by titration either with potassium ferrocyanide, or with ammoniacal sodium sulphide. The author points out that the solution finally titrated in the usual ferrocyanide method may contain cadmium and manganese, both of which are precipitated by ferrocyanide, and therefore returned as zinc; the cadmium content is generally too low (0.1 per cent.) to influence results, but manganese may be present to the extent of 1 per cent. or more. Manganese can be eliminated by adding a few c.c. of hydrogen peroxide to the mixed ammonia and ammonium chloride solution used to treat the residue obtained by the decomposition of the ore or concentrate with nitric acid and chlorate. The ferrocyanide method has the disadvantage that the solution has to be titrated hot, whilst the blue colour of the spot test is not always very distinct in presence of coloured ferrocyanides. In the author's opinion the sulphide method is preferable, especially as the zinc is precipitated as a colourless sulphide, which affords indication of its freedom from metallic sulphides other than cadmium.

R. G. P.

Approximate Estimation of Iron and Manganese. L. J. Curtman and N. H. Hecht. (*Chem. News*, 1921, **122**, 254-255.)—A precipitate containing

aluminium and chromium hydroxides, and nickel, cobalt, iron, manganese and zinc sulphides, is treated with hydrochloric acid, the insoluble nickel and cobalt sulphides are separated by filtration, the filtrate is then evaporated to a small volume, and diluted to 50 c.c. Twenty-five c.c. of this solution are boiled with the addition of sodium hydroxide and sodium peroxide; the precipitate is collected, dissolved in hydrochloric acid, the solution treated with potassium thiocyanate solution, and then titrated with standardised stannous chloride solution until the pink coloration matches that obtained by adding thiocyanate to 0.05 mgrm. of iron in 50 c.c. of dilute hydrochloric acid. It is not convenient to continue the titration to a colourless end-point owing to the slowness of the final reaction. Another portion of 25 c.c. of the solution is treated as described, the precipitate produced by the peroxide treatment is washed free from chlorides, dissolved in nitric acid, the solution diluted to a definite volume, and an aliquot portion treated with sodium bismuthate; the permanganate produced is titrated with hydrogen peroxide solution.

W. P. S.

Detection and Estimation of Cobalt. S. A. Braley and F. B. Hobart. (*J. Amer. Chem. Soc.*, 1921, **43**, 482-484.)—In the absence of elements which give coloured solutions or precipitates with sodium acetate or dimethylglyoxime (with the exception of nickel), the brown colour of cobalt dimethylglyoxime is proportional to the concentration, and affords a basis for the colorimetric estimation of this metal. The solution containing cobalt and nickel, prepared as for the estimation of nickel, is slightly acidified, 10 per cent. sodium acetate solution is added, the mixture boiled and treated with an excess of dimethylglyoxime. Nickel is separated by filtration, the filtrate cooled, and the coloration compared with that of a standard containing a known amount of cobalt.

W. P. S.

Sodium Salicylate as Reagent for Detecting Aluminium and Manganese. C. van Zijp. (*Pharm. Weekblad*, 1921, **58**, 694-698.)—When solutions of sodium salicylate and aluminium nitrate are caused to react, a microscopic examination reveals the formation of colourless needles which tend to coalesce into spherical groups. With excess of aluminium solution, a granular mass is first formed and development of crystals is retarded, whilst excess of salicylate dissolves the reaction product. Addition of caesium chloride assists the reaction, and detection of the crystals is further facilitated by exposing the drop of the mixed solutions to the action of ammonia vapour, by which any precipitated hydroxide is dissolved. The method is suitable for detecting aluminium, iron, and chromium, either alone or in presence of each other; the solutions must be of such concentration as to prevent formation of crystals before treatment with ammonia, and, if any hydroxides are precipitated, the dried residue must be carefully treated with a drop of water, which is then separated and again allowed to evaporate. Iron salts give a violet coloration, changing to yellow after treatment with ammonia, but no hydroxide is precipitated. On the other hand, ammonia precipitates chromium hydroxide from chromium salts, and from mixtures of iron and chromium salts hydroxides of both metals are formed. Crystals are only produced when aluminium salts are

present. If the aluminium solution contains iron as well, the crystals show a brown coloration, but no hydroxides are precipitated; in presence of chromium, on evaporation of the drop there is considerable formation of hydroxides, but the crystals obtained after treatment with water are colourless. The presence of both iron and chromium in the aluminium solution is indicated by separation of mixed hydroxides with a yellow colour, and by the brown colour of the crystals. Manganese solutions, when mixed with sodium salicylate, do not crystallise, but when the mixture is exposed to the action of ammonia, light green, pointed crystals are formed. If zinc be present in the manganese solution, a slight excess of sodium salicylate must be employed, and the mixed solutions are then treated with ammonia vapour until any hydroxides formed redissolve; such solution will not occur if too little salicylate was used in the first case.

W. J. W.

Estimation of Phosphorus in Phosphor-Bronze. G. Greenwood. (*Chem. News*, 1921, **122**, 225-226.)—A mixture of 1 grm. of the alloy and 0.5 grm. of iron wire or mild steel borings is dissolved in 15 c.c. of *aqua regia*, the solution boiled for a few minutes, cooled, and nearly neutralised with ammonia. Potassium permanganate is then added in quantity sufficient to produce a distinct precipitate when the mixture is boiled for two minutes, the precipitate is dissolved by the addition of a few drops of hydrochloric acid, the solution is cooled, diluted, and treated with 25 cc. of ammonia (sp. gr. 0.880). The precipitated basic ferric phosphate is dissolved by the addition, drop by drop, of concentrated nitric acid, an excess of 3 c.c. of nitric acid is added, the mixture boiled, and the phosphoric acid precipitated by the addition of molybdic acid reagent. After twenty minutes, the yellow precipitate is collected, washed with 2 per cent. nitric acid, then with 2 per cent. potassium nitrate solution, dissolved in a known excess of standard sodium hydroxide solution, and this excess titrated with standard nitric acid, using phenolphthalein as indicator. The solutions should be standardised against a steel of known phosphorus content; an allowance is made for the small amount of phosphorus in the added iron wire or mild steel.

W. P. S.

Electrometric Titration of Hypochlorous Acid. W. D. Treadwell. (*Helv. Chim. Acta*, 1921, **4**, 396-405.)—The end-point of the titration of hypochlorite with arsenious acid (Penot, 1851) may be determined accurately by electrometric means. As comparison electrode, use is made of a glass tube about 8 cm. long, with its end drawn out to a capillary and bent upwards, the tip of the capillary being closed in the ordinary way with a plug of filter-paper or a drop of gelatin containing potassium sulphate. The tube is filled with potassium sulphate solution to which has been added either a little hypochlorite solution already titrated, or a drop of about 0.01 *N*-iodine solution containing iodide. Two smooth platinum wires serve as electrodes, one dipping into the hypochlorite solution, and the other fairly deeply into the comparison electrode. The two electrodes are connected either through a high-resistance millivoltmeter, or through a galvanometer with suitable resistance. The end-point of the titration is indicated by a sudden fall of the galvanometer deflection

to zero. Titration of an alkaline hypochlorite with arsenious acid must be carried out immediately after the neutralisation of the solution; if the titration is effected in dilute solution at the ordinary temperature and within a few minutes of neutralisation, the losses due to premature formation of chlorate remain immeasurably small. In the case of solutions containing alkali carbonate or hydroxide this titration should not be applied, the end-point then losing in sharpness.

As regards titration of hypochlorite with potassium iodide, the optimum acidity for the end-point of the titration may be calculated from the equilibrium, $\text{IO}_3' + 5\text{I}' + 6\text{H} \rightleftharpoons 3\text{H}_2\text{O} + 3\text{I}_2$, which has been thoroughly investigated. At 25°C . the relation $\frac{(\text{H})^6 \cdot (\text{IO}_3') \cdot (\text{I}')^5}{(\text{I}_2)^3} = 2.8 \times 10^{-46}$ holds. During the titration (H) is virtually constant, and at the end of the titration (IO_3') may be regarded as constant, and it has been shown that in iodide of starch the ratio $(\text{I}_2) : (\text{I}')$ has the value 2. From these considerations it follows that the optimum acidity of the end-point is represented by $(\text{H}) = 10^{-7}$, which is that of the neutral point. The great sensitiveness of the end-point to acid constitutes the essential weakness of this method, and Pontius (1904), to whom the method is due, employed a too highly alkaline solution [$(\text{H}) = 10^{-9}$], resulting in a retardation of the end-point. If the available chlorine amounts to about 0.1 equivalent per litre, a solution alkaline with bicarbonate yields satisfactory results, but with 0.01 *N*-hypochlorite the results are uncertain. If, with a highly dilute solution, the final stage of the titration be not carried out very slowly, the blue coloration of the starch appears far too late. Here, too, the end-point may be determined electrometrically, but in any case the method is less exact and less sensitive than titration with arsenious acid.

T. H. P.

Separation and Detection of Arsenate and Arsenite. G. W. Sears. (*J. Amer. Chem. Soc.*, 1921, **43**, 466-470.)—The method depends on the solubility of silver arsenate and on the insolubility of silver arsenite in dilute sodium hydroxide solution; the concentration of the latter should not exceed 1.5 *N*. A nitric acid solution containing an arsenite and arsenate and an excess of silver nitrate is treated with sodium hydroxide solution until the dark silver oxide precipitate just fails to redissolve; the precipitate is collected, washed, rinsed into a graduated cylinder with 20 c.c. of water, 3 c.c. of 6 *N* sodium hydroxide solution are added, and the mixture is diluted to 25 c.c. The amount of sodium hydroxide required depends on the quantity of arsenate present; 3 c.c. of 6 *N* sodium hydroxide solution in 25 c.c. of total liquid will dissolve more than 0.45 gm. of arsenate, whilst 5 c.c. of the alkali solution in the same volume of liquid has no apparent solvent action on the arsenite. The contents of the cylinder are mixed, filtered, and the insoluble portion washed until free from alkali. *Detection of Arsenite*: The insoluble portion is dissolved in a small quantity of dilute hydrochloric acid and the solution tested with hydrogen sulphide; if the sulphide precipitate has a dark colour, heavy metals are present, and must be separated by the usual methods. *Detection of Arsenate*: The filtrate is acidified with acetic acid, uranyl acetate is added in slight excess, the precipitated uranyl hydrogen arsenate is collected, dissolved in hydrochloric acid,

and the arsenic precipitated with hydrogen sulphide. The method is capable of detecting 1 mgrm. of either ion in the presence of 450 mgrms. of the other.

W. P. S.

PHYSICAL METHODS, APPARATUS, ETC.

New Technique for Microscopical Preparations of Vegetable and Animal Tissues. Larbaud. (*Comptes rend.*, 1921, 172, 1317-1319.)—The ordinary method of preparing fresh tissues to be imbedded in paraffin wax consists in treating them successively in baths of aqueous alcohol of increasing concentration, followed by two mixtures of alcohol and xylene (or toluene) and then by pure xylene. This procedure is disadvantageous, owing to the presence of impurities in the reagents, to the presence of traces of water in so-called absolute alcohol, to the contraction of the protoplasm and the hardening of the tissues, and to the costliness of the solvents employed. A mixture of normal butyl alcohol with 95 per cent. (by volume) ethyl alcohol is preferable. For the first four baths, 100 c.c. of this mixture is mixed with 225 c.c., 62.5 c.c., 21.87 c.c., and 2.63 c.c. of water respectively. The treatment is completed by means of two baths of the butyl alcohol, to the second of which paraffin wax is added little by little; the subsequent operations are as usual. Specimens thus treated become neither hard nor brittle, whilst the protoplasm undergoes no contraction.

T. H. P.

Spectrum Analysis by Means of Roentgen Rays. M. Siegbahn, A. E. Lindh, and N. StenSSon. (*Zeit. Physik.*, 1912, 4, 61-67; *J. Chem. Soc.*, 1921, 120, ii. 344.)—A vacuum spectrograph enables the X-ray spectrum to be employed for spectrum analysis of mixtures in the usual course of analysis. The instrument has an opening angle of about 40° and a fixed crystal of calc spar or gypsum, making it possible to obtain with two exposures of about two hours each photographs of the lines of all elements from uranium to sodium.

R. G. P.

Check Valve for Water Vacuum Pumps. E. A. Andelin. (*Chem. Zeit.*, 1921, 56, 448.)—The check valve described by Pinoff (*Chem. Zeit.*, 1920, 671) is not absolutely reliable, as it does not always function instantaneously. An improved device comprises a rubber bung, 10 to 12 mm. diam., with a central perforation of 5 mm. diam., through about two-thirds of its length. A transverse cut is made in the bung so as to separate the unbored portion and form a flap valve, and the whole is fitted on a glass tube. By means of another rubber bung the device is fixed in a second tube of 20 mm. diam., which is drawn out at its lower end. The apparatus should always be used in a vertical position.

W. J. W.

Slide Rule for Calculation of "Wet" and "Dry" Weights of Damp Material. R. Scott. (*J. Soc. Chem. Ind.*, 1921, 40, 96r.)—A slide rule which will show the amount of "wet" material required to give a specific amount of "dry" material, and *vice versa*, may be constructed by dividing the frame scale in the usual logarithmic manner, and the slide scale proportionally to $\log 100/(100 - \text{moisture per cent.})$. It is convenient to make the distance from 10 to 100 on the frame

1,000 mm. long, and to extend the frame beyond 100 to a length equal to that of the slide scale. If then the zero mark on the slide scale be placed opposite the division of the frame corresponding to the "dry" weight, the wet weight is read off opposite the division on the slide scale corresponding to the moisture content. For calculating dry weight the procedure is reversed. The results should be accurate to within 0.5 per cent.

H. E. C.



DANGEROUS DRUGS ACT.

REVISED REGULATIONS.

In pursuance of Section 3 of the Dangerous Drugs Act, 1920, the Home Secretary has made the following Regulations, the full text of which is published in the *London Gazette* of May 24, 1921. These Regulations come into force on September 1, 1921.

Opium : (1) to (3) No person shall supply, procure, or be in possession of raw opium, unless licensed by the Secretary of State, or authorised by these Regulations. (4) Records in specified form shall be kept of all dealings in raw opium.

General Authorisations.—(5) Any duly qualified medical practitioner, or any person lawfully keeping open shop for the retailing of poisons in accordance with the provisions of the Pharmacy Act, 1868, as amended by the Poisons and Pharmacy Act, 1908, or any person employed or engaged in dispensing medicines at any public hospital or other public institution being a person duly registered under the Pharmacy Act, 1868, as amended by the Poisons and Pharmacy Act, 1908, or any registered veterinary surgeon or any person in charge of a laboratory for purposes of research or instruction attached to any University, University College, public hospital, or other institution approved by the Secretary of State for the purpose, or any person appointed by a local authority with the approval of the Minister of Health as an analyst for the purposes of the Sale of Food and Drugs Acts, 1875 to 1907, is hereby authorised so far as is necessary for the practice of his profession or employment in such capacity to be in possession of and supply raw opium, but subject always to the provisions of the foregoing Regulation. (6) Licences will be withdrawn for offences against the Act. (7) No raw opium is to be delivered to messengers unless written authority from a licensed person is produced. (8) "Possession" means at the order or disposition of a person.

Morphine, Cocaine, Ecgonine, and Diamorphine : *Application.*—(1) The drugs to which these Regulations apply are morphine, cocaine, ecgonine, and diamorphine (commonly known as heroin), and their respective salts, and medicinal opium, and any preparation, admixture, extract, or other substance containing not less than one-fifth per cent. of morphine or one-tenth per cent. of cocaine, ecgonine, or diamorphine. For the purpose of the foregoing provision, the percentage in the case of morphine shall be calculated as in respect of anhydrous morphine. (2) Prohibits the manufacture of these drugs, except by licensed persons on licensed premises. (3) and (4) Prohibit the sale, distribution, or possession of the drugs, except by licensed persons or those authorised by the Regulations. (5) and (6) Give the conditions for the prescription and dispensing of the drugs by authorised persons. (7) Prohibits possession except by licensed or authorised persons. (8) Requires packages to be marked with the amount of drug contained in them, except in the case of authorised prescriptions. (9) Records must be kept in specified form.

General Authorisations.—(10) Any person lawfully keeping open shop for the retailing of poisons in accordance with the provisions of the Pharmacy Act, 1868, as

amended by the Poisons and Pharmacy Act, 1908, is hereby authorised—(a) To manufacture at the shop in the ordinary course of his retail business any preparation, admixture, or extract of any of the drugs; (b) to carry on at the shop the business of retailing, dispensing, or compounding the drugs, but subject always to the provisions of these Regulations. (11) to (14) are the same as (5) to (8) in the Opium Regulations. (15) Gives the Regulations affecting the captains of ships not carrying a medical practitioner. (16) Hospitals or other public institutions may be exempted from the operation of the Regulations.

Preparations Exempted from the Regulations.—(17) These Regulations shall not apply in respect of the preparations named in Schedule II. to these Regulations, nor to any of the drugs when denatured in a manner approved by the Secretary of State. The Secretary of State may from time to time by order add any other preparation to the Schedule, or remove any preparation from the Schedule.

SCHEDULE II.

Cereoli Iodoformi et Morphinæ, B.P.C.	Pil. Hydrarg. c. Cret. et Opii, B.P.C.
Emp. Opii, B.P., 1898.	Pulv. Cretæ Aromat. c. Opio, B.P.
Lin. Opii, B.P.	„ Ipecac. Co., B.P. (Dover's Powder).
„ Opii Ammon., B.P.C.	„ Kino Co., B.P.
Pasta Arsenicalis, B.P.C.	Suppos. Plumbi Co., B.P.
Pil. Hydrarg. c. Opio, B.P.C.	Tablettæ Plumbi c. Opio, B.P.C.
„ Ipecac. c. Scilla, B.P.	Ung. Gallæ c. Opio, B.P. (Gall and Opium
„ Plumbi c. Opio, B.P.	Ointment).
„ Digitalis et Opii Co., B.P.C.	„ Gallæ Co., B.P.C.

FOOD CONTROL.

STATUTORY RULES AND ORDERS.

No. 350, 1921.

THE MILK (ENGLAND AND WALES) ORDER, 1921, DATED MARCH 22, 1921, MADE BY THE FOOD CONTROLLER UNDER THE MINISTRY OF FOOD (CONTINUANCE) ACT, 1920 (10 AND 11 GEO. 5, c. 47).

1. EXCEPT under and in accordance with a licence granted by or under the authority of the Minister of Health, a person shall not—(a) Sell or expose for sale any milk as “Grade A (certified)” or “Grade A” milk; or (b) describe or refer to any milk under these names in any advertisement, circular, or notice in connection with the sale of milk; or (c) sell or offer or expose for sale as milk, or under any description of which the word “milk” forms part, any liquid in the making of which dried milk or condensed milk has been used.

2. A licence may be granted under this Order for such period and subject to such terms and conditions as the Minister of Health may think fit.

3. No colouring matter or water shall be added to milk or cream intended for sale, and no milk or cream to which any colouring matter or water has been added shall knowingly be sold or offered or exposed for sale.

4. (a) Every local authority is hereby authorised to execute and enforce the provisions of this Order within their area, and to institute proceedings for any offence against this order. (b) For the purposes of this clause “local authority” means any local authority authorised to appoint an analyst for the purposes of the Sale of Food and Drugs Acts, 1875 to 1907.

5. For the purposes of this Order, “milk” shall include any milk which has

been submitted to one or more of the following processes—viz., sterilisation, pasteurisation, homogenisation, or any other like process, but shall not include condensed, separated, skimmed, or butter milk.

6. The Milk Order, 1920, is hereby revoked, so far as it applies to England and Wales, as on March 24, 1921, but without prejudice to any licences granted thereunder and in force at the date of this Order.

7. (a) This Order may be cited as the Milk (England and Wales) Order, 1921, and shall come into force on March 24, 1921. (b) This Order applies only to England and Wales. (c) The Interpretation Act, 1889, applies to the interpretation of this Order as it applies to the interpretation of an Act of Parliament.

No. 379, 1921.

THE LOCAL AUTHORITIES (MILK) ORDER, 1921, DATED MARCH 24, 1921, MADE BY THE MINISTER OF HEALTH.

THE Minister of Health by arrangement with the Food Controller orders as follows :

1. (i.) Every local authority authorised to appoint an analyst for the purposes of the Sale of Food and Drugs Acts, 1875 to 1907, shall have all such powers and duties as may be necessary for the execution within their area of the functions assigned to them by the Milk (England and Wales) Order, 1921.

(ii.) Every local authority as aforesaid, and any officers of the local authority duly authorised in that behalf, may at all reasonable times enter and inspect any premises in which milk sold or offered for sale in accordance with a licence given under the Milk (England and Wales) Order, 1921, is produced or prepared for sale.

2. Any expenses incurred by a local authority as aforesaid in the execution of this Order shall be defrayed in the same manner as expenses of the local authority are defrayed for the purposes of the Sale of Food and Drugs Acts, 1875 to 1907.

3. This Order may be cited as the Local Authorities (Milk) Order, 1921.

No. 406, 1921.

THE MILK (SCOTLAND) ORDER, MARCH 31, 1921, MADE BY THE FOOD CONTROLLER UNDER THE MINISTRY OF FOOD (CONTINUANCE) ACT, 1920.



REVIEWS.

THE ELECTRONIC CONCEPTION OF VALENCE AND THE CONSTITUTION OF BENZENE.
By H. S. FRY. Monographs on Inorganic and Physical Chemistry; edited by
Alexander Findlay. Pp. xviii + 300. London: Longmans, Green and Co.
1921. Price 16s. net.

The subject of valency and valence is one in which English chemical literature is sadly deficient, so that the present volume is a most useful and welcome addition; the more so as most of the theories of valence have been expounded and discussed in foreign journals. We have now presented to us in a most lucid manner by Professor Fry a detailed account of the electronic theory of valence, which, even in the Annual Reports of the Chemical Society, has not received much notice in this country. Professor Cohen, in his "Organic Chemistry" (vol. i. p. 161), remarks that "the theory is so mobile, so adaptable, and so ingeniously applied as to explain most of the facts of substitution as well as many of the reactions of organic compounds." One cannot read this fascinating volume without being impressed with the truth of this statement; the manner in which the hitherto most baffling problems in organic reactions are explained in the light of a single fundamental conception of the chemical bond as a unit Faraday tube connecting charged atoms is quite remarkable. The theory put forward, only as a formulative hypothesis, is developed so as to account rationally for not only the problems of substitution in the benzene nucleus, but the whole range of organic chemistry, including such diverse branches as velocity of substitution, absorption spectra, physical properties, molecular volumes, and fluorescence. Even the complexities of the structure of the metal-aminines are smoothed out by the skilful treatment of the author, and discussed with mathematical exactness without the introduction of speculative assumptions or vague statements. The fact that the electronic theory can be successfully applied over such a wide range of experimental results places it above any other theory of valence extant; it certainly maintains its inventor's modest claim that it should function as a formulative hypothesis in the interpretation and correlation of chemical and physico-chemical phenomena.

The book, in its presentation and detail, maintains well the excellent standard of Professor Findlay's series of monographs. The style is lucid and exact, the development of the subject is quite fascinating, and there is an almost complete freedom from typographical errors. The table of contents is very full, but a subject

index at the end would be a useful adjunct for reference purposes, and the table of references to other literature might be a little extended, as there are a few places (*e.g.*, pp. 18 and 144) where quotations are made and the reference is not given.

Probably the advocates of alternative theories of valence will find points in the arguments which may be debated and controverted, but the book is undoubtedly a most valuable contribution to modern chemical theory, and one on which the author, editor, and publishers may well be complimented.

H. E. Cox.

THERMODYNAMICS AND CHEMISTRY. By F. H. MACDOUGALL, M.A., Ph.D. John Wiley and Sons. Pp. v+391. London: Chapman and Hall, Ltd. Price 30s. net.

The outstanding feature of Professor Macdougall's book may be said to be thoroughness. No pains are spared in the definition and explanation of every term used, and in the explanation as well as the rigid proof of the most elementary of the thermodynamical deductions. Throughout the book stress is laid upon the empirical nature of such doctrines as the First and Second Laws of Thermodynamics, and also the fundamental chemical laws, the Atomic Theory and Avogadro's Hypothesis. The First and Second Laws are treated at some length, and thence the questions of entropy and the thermodynamical scale of temperature, which are considered in detail.

The treatment of the Phase Rule is exhaustive both in proof and in illustration, and the various types of phase system are considered minutely. A point that might well be copied is the inclusion with each type of phase diagram of a list of examples of the various systems belonging to the type illustrated. The increasingly popular system of triangular co-ordinates is well illustrated in some examples of ternary systems. The excellent section on fractional distillation and on partially miscible liquids should be of particular value to the organic chemist.

The laws of osmotic pressure are deduced as applications of the Phase Rule—rather a departure from usual practice. Chapter XVI., which contains some ionisation equations, necessary but not strictly thermodynamical, comprises a useful section on Activity, and a summary of the equations expressing the behaviour of strong electrolytes. Electromotive Force is dealt with in a comprehensive and complete manner. In view of the increasing part played in chemical theory by surface tension and adsorption, Chapter XVIII. is of great interest.

The final chapter is a triumph of conciseness and thoroughness. Within thirty-three pages it embraces the Laws of Radiation, the Quantum Theory, Debye's Theory of Atomic Heat, and the Nernst Heat Theorem and some of its applications, and concludes with a set of tables of numerical values of the Einstein and Debye functions. We might well wish this chapter to be amplified and subdivided in order to give a greater number of examples of the application of these most important principles.

Altogether the book is a masterly treatment of thermodynamics and its bearing on chemistry, and should commend itself equally to the chemist and to the physicist.

H. R. AMBLER.

INTRODUCTION TO QUALITATIVE CHEMICAL ANALYSIS. By TH. W. FRESenius. Seventeenth Edition of the Original Work by C. R. FRESenius. Translated by C. A. MITCHELL, M.A., F.I.C. Pp. 954. London: J. and A. Churchill. 1921. Price 36s. net.

The appearance of a new English edition of Fresenius is an event of very much more than passing interest to the great body of professional chemists in all countries in which the English language is spoken.

Despite the fact that the production of Fresenius was planned as long ago as 1840, and that, nearly fifty years ago, nine English and fourteen German editions had already appeared, this work still remains the standard textbook of general analytical chemistry—a book which the young student may take as his safest guide, and to which, at the same time, the experienced practitioner may confidently turn for help and sound advice in cases of uncertainty and difficulty.

To attempt anything in the nature of detailed criticism of a work of this character would obviously be a task beyond the power of any one reviewer, and, even if it were possible, it would be a work of supererogation, for the best testimony to its value and its accuracy is to be found in the esteem in which it is still held by many thousands of chemists in all parts of the world, who during many years have, like their predecessors, tested it thoroughly, and have very rarely found it wanting.

One's first impulse, on the appearance of a new edition of a classic such as this, is to compare it with previous editions, for the purpose of seeing what new features have been introduced, and of ascertaining to what extent the work has kept pace with the progress made in the subject.

The edition on which the writer of this review was brought up was the ninth English, corresponding with the fourteenth German edition, published in 1876.

As a reply to those persons who have, on occasions, foolishly expressed the opinion that analytical chemistry represents a branch of the science in which little further progress can be expected, it may be pointed out that, although the volume under review deals with qualitative analysis alone, and is written in a condensed style, the number of pages has increased from 400 in 1876 to 950 in the present volume.

In the Preface to the 1876 edition, the following statement by the author, in reply to a letter of remonstrance from the late Sir Edward Frankland, is of interest: "I do not retain the old notation from prejudice, but from a most firm conviction that, for inorganic chemistry, it is the simplest and the best. This view is not peculiar to myself alone, but is shared with the highest authorities in Germany—Wohler and Bunsen; Professor Kopp also, one of the first representatives of theoretical chemistry, had advised me decidedly not to relinquish my notation."

In the present volume, not only the notation, but the whole presentation of the subject, are in accordance with the most recent chemical and chemico-physical theories, and in this respect the book may certainly be said to have kept abreast of the developments of the science.

Notwithstanding the very great improvement which has been made during recent years in the status of analytical chemistry, there is still a rather widespread

impression that it is a useful art or, at the best, an inferior branch of the science, and Professor Dittmar, in his admirable little work on "Qualitative Chemical Analysis," published in 1876, put the matter very clearly when he said:

"All we desire is, at the very outset, to make the student understand that analysis is not an art which can be learned independently of chemistry itself. It is possible to be a fair theoretical chemist without being able to execute an analysis, but most assuredly no one can be an analyst without being a scientific chemist."

If that was true—as it undoubtedly was—in Dittmar's time, it is more than ever true to-day, and this view has clearly been uppermost in the mind of the author of the work under review.

Full, clear, and, in the main, adequate descriptions are given of the fundamental conceptions of chemistry, as well as of the general principles underlying all the various procedures with which the analyst is concerned. At the same time, the author has avoided the danger of overloading the book with matter for which the student must in any case consult general systematic treatises.

So far as the reviewer can see, the work has been brought thoroughly up to date. Among other things, much new matter in relation to the reactions of the rarer elements has been added, and the many reactions in which organic substances are employed as reagents are accurately and adequately described.

The only serious criticism which the writer has to make is one which can be made of most German scientific works—namely, the failure of their authors to refer to original sources other than German. Had the author of this work avoided this national failing, one or two sections would certainly have gained in value.

On what principle the properties of the alkaloids are given in a lengthy appendix, to the exclusion of other poisonous organic compounds, is not very clear, especially as the information is to be found in all treatises on general toxicology.

The writer has observed only a few typographical errors, and the printing and paper are all that could be desired.

The translation is good, and the translator, Mr. C. A. Mitchell, is to be warmly congratulated on the successful manner in which he has accomplished a somewhat difficult task.

For the sake of old associations, if for no other reason, one is glad to see that the coloured frontispiece representing metallic spectra is retained. Without it Fresenius would scarcely be Fresenius.

A. CHASTON CHAPMAN.

APPLIED COLLOID CHEMISTRY—GENERAL THEORY. By WILDER D. BANCROFT, Ph.D., Professor of Physical Chemistry at Cornell University, U.S.A.. Pp. 345. London: McGraw-Hill Book Co. Inc. Price 18s. net.

The importance of colloid chemistry for the solution of ubiquitous industrial problems is being more and more recognised. Propaganda work is being carried on by committees of experts on colloid chemistry in England and in the United States of America. Foremost amongst the American authorities is Professor Bancroft, and

his book, part of the general scheme of the American programme, is very welcome indeed.

His treatment of the subject differs from the usual textbook on colloid chemistry. In his Preface he says: "The earlier books on colloid chemistry presented the subject empirically, because no other method was then possible. While we do not know much about gelatinous precipitates and jellies, the theory of the rest of the subject is in fairly good shape, and consequently I have written this book deductively. While my point of view differs considerably from Freundlich's, it is a pleasure to acknowledge my debt to him for what he did in his admirable book."

Professor Bancroft makes out a good case for treating colloids from the standpoint of adsorption, a field in which we now possess more definite *quantitative* data than in other fields of colloid phenomena. He shows how adsorption must be taken into account in connection with surface tension effects, coalescence, precipitation, peptisation, the properties of colloidal solutions, emulsification, and fogs and smokes. The discussion is well presented, and is far from being a mere statement of facts. There is a welcome critical vein throughout, new problems for research are suggested, and obvious difficulties in the acceptance of certain deduced propositions are not shirked.

The chapter on emulsions and foams is good and up to date. Colloid textbooks so far have only dealt with the subject of emulsions in most meagre fashion, so that students will thank the author for his summary in this field. New, also, are the discussions on colloids in relation to catalysis, colour, and fogs and smokes. The closing chapter deals with the thickness of surface films, and is a useful summary of the work done, though a discussion of the new views on the orientation of molecules in liquid surfaces and their relation to colloid phenomena would have been welcome. One notable omission from this excellent book is that of Donnan's theory of membrane equilibria, to which Loeb has recently announced his ardent support, as a result of his own extensive investigations, particularly on gelatin.

This book is well bound, and the paper and printing are also good. Several typographical errors are apparent, among them an error in the equation on p. 141, but they do not detract from the value of a refreshingly up-to-date and readable textbook on the general theory of colloid phenomena.

WILLIAM CLAYTON.

LUBRICATING AND ALLIED OILS. By ELLIOTT A. EVANS. Pp. 128. London: Chapman and Hall, Ltd. 1921. Price 9s. 6d. net.

In perusing this book, it is difficult to decide exactly what ground the author intended to cover. The title includes lubricating and allied oils, whatever that may mean; but in the Foreword by Sir Charles Wakefield lubricating oils alone are mentioned, and in the Preface the author says he wrote the book "after repeated requests from chemists, engineers, and others for a useful non-too-technical book on lubricating oils . . . with a view to assist chemists in compiling specifications and examining lubricating oils; also to give engineers an insight into the application and properties of such oils and the interpretation of their specifications." He curiously complains that existing treatises are "too technical, except for the specialist or person

who desires knowledge on a specific section of the subject." He admits that chemists require detailed information on the methods used in the laboratory, which he says some of the books do not provide. One is curious to find how this kind of information is to be given in a "non-too-technical" book, and one finds that when the author sets out to seriously describe or discuss a test or method he has either to be as technical as any other author or to slur over the subject.

Another point one notices is that, whilst the author says he has endeavoured to include only those tests which are commonly demanded, he has gone into quite a lot of technical detail regarding tests of quite minor importance, such as the determination of specific heat, with tables of the specific heats of a number of pure hydrocarbons, occupying seven pages of this small book. One also finds, as foreshadowed by the title, that the author has included a number of tests used in the examination of fuel oils and transformer oils, which do not concern lubricating oils. One finds in the book very little real guidance in the framing of specifications or in their interpretation, and in the chapter on the oils employed for various purposes three pages contain a list of proprietary grades of Wakefield's "Castrol" suitable for the various types of motor vehicles, which is, of course, pure advertisement. No reference whatever is made to the value of a little free fatty acid in improving the friction-reducing qualities of mineral oils. We are told, for instance, on p. 110 that "locomotive engineers in this country call for an oil containing anything from 5 to 75 per cent. of rape oil, according to their needs, in the mineral oil"; but the author does not advise whether this is good or bad practice, whether such variation in the proportion is necessary, and whether, in fact, the whole or the greater part of this rape oil might not be saved and a little fatty acid used instead. Few locomotive engineers now use anything like so large a proportion as 75 per cent. of rape oil in the lubricant.

In the first three chapters, occupying only seven and a half pages, the author gives a scrappy but interesting account of the history of petroleum, and very brief remarks on oil refining and on the fatty oils. "Ravine rape" at the foot of p. 9 presumably refers to the oil usually known as ravisin oil. In Chapter IV., on Physical Tests, the author shows how the presence of water in an oil may influence the flash-point, but "does not favour the drying of the oil before making a test, because he believes it detracts from the value of the test." Whether it does or not would seem to depend upon whether the operator wishes to ascertain the flash-point of the oil or has some other object in view. The author remarks that, "pending a better system of classification, lubricating oils are generally grouped according to their viscosity." Seeing that viscosity is one of the most important properties of a lubricant, one wonders what better system of classification the author has in his mind. As an example of loose phraseology, the author states on p. 30 that in the Engler, Redwood, and Saybolt viscometers "the greater portion of the energy of flow is lost in the rapid passage of the liquid through the tube." What is probably meant is that when thin liquids are being tested the greater part of the energy due to the head appears as energy of flow—i.e., kinetic energy—instead of being converted into heat by the viscosity, as the author shows in the sentence following that from which the quotation is taken. It is time that the viscosity values of oils were expressed in absolute

measure or in units, such as the centipoise, which are independent of the instrument used in determining them, but the author seems to have no definite views on the subject, and to be quite content with the present system.

Those chemists who have to advise on lubricating and other oils will find useful information in this book, but it cannot, in the reviewer's opinion, be regarded as a very valuable addition to the literature of the subject.

L. ARCHBUTT.

FAMOUS CHEMISTS: THE MEN AND THEIR WORK. By Sir WILLIAM A. TILDEN, F.R.S., D.Sc. London: George Routledge and Sons, Ltd. Price 12s. 6d. net.

Sir William Tilden, in his Preface, disclaims for his book the character of another history of chemistry, but thinks he is justified in offering to the public this volume of biographical essays dealing with well-known chemists, because other similar works have not been contributions to the history of chemistry. All chemists will be indebted to him for his book. It was a good idea to make use of the atomic theory as the string on which to thread his biographical beads, of which there are twenty-one divided into ten groups. The groups are appropriately named to mark the epochs in the history of the theory of chemistry.

The author anticipates criticism with regard to the names which he has felt himself compelled to reject. Those which he has selected for treatment will, one feels sure, commend themselves to all those who have regard to the fact that the available space in the book is only about three hundred pages. Sir William's own career would lead his readers to expect that any such book as this written by him would at least subserve a didactic purpose in the field of chemical theory. It does this most admirably, and, let it be gratefully added, most agreeably.

Robert Boyle is the subject of the first essay, which is a quite satisfactory account of "the father of chemistry," inasmuch as that, in addition to the compendious biographical matter, particulars are given as to the experiments establishing the "Law of Boyle"; and it is indicated what Boyle did in bringing the conception of an *element* out of the realms of philosophy and pseudo-philosophy into the realm of matter. The author has most significantly succeeded, without using any undue emphasis, in displaying the development of the conception of the element, considered from the atomic point of view, from the purely speculative stage, through the material stages, right down to the physical conception of ultra-chemistry of the present day.

After Boyle comes the second group of "The Phlogistians," comprising Black, Priestley, Cavendish, and Scheele. Scheele has been dealt with most sympathetically. Lavoisier is taken as the representative of the antiphlogistic revolution. The article on Davy is one of the longest and probably the best, and it would lead one to think that Davy is one of Sir William's favourites. As a presentation of the character of this great man it is, on the whole, rather too favourable. It is only on reading a subsequent essay on Berzelius that one learns, from the remarks of Berzelius on the character of Davy, that Sir William is aware that the impression produced by Davy on his contemporaries was not consonant with either his position in the world of science or with his undoubted abilities.

On the whole, the subjects of the book are treated so as to produce the feeling

that they are altogether in the hands of a warm admirer rather than in the hands of a historian. In the case of Cavendish there is only the barest reference to the "water" controversy. It is matter for commendation that the author has written so excellent an account of Berzelius and his work, and attempted to do justice to his memory as a great and widely influential teacher. Space forbids the dealing at length with features of the other essays which treat of Dalton, Gay-Lussac, Proust, Faraday, Avogadro, Cannizzaro, Liebig, Dumas, Frankland, Williamson, Mendeléef, Crookes, and Ramsay.

The book contains nearly forty illustrations, and there is at least one portrait of each chemist mentioned. The printing and paper are good, and the proof-reading has been pretty well done; on p. 105 Dalton's birth year is given as 1706 instead of 1766, and there are less than half a dozen other "literal" errata. The volume is in every respect an admirable one for the chemist and for the general cultured reader.

W. KIRKBY.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

NOTES ON THE MEASUREMENT OF HYDROGEN ION CONCENTRATION.

By G. W. MONIER-WILLIAMS, O.B.E., M.C., M.A., F.I.C.

(Read at the Meeting, May 4, 1921.)

THE determination of absolute acidity or hydrogen ion concentration has not yet found its way into many analytical laboratories, mainly on account of the expense of the apparatus which is usually considered necessary. The object of this paper is to indicate how the electrical measurement of acidity can be carried out with comparatively simple and inexpensive apparatus with a degree of accuracy comparable to that attained by titration methods.

THEORETICAL BASIS OF THE METHOD.—The elementary theory of the method can be stated quite briefly. If a rod of metal be dipped into water, a minute quantity of the metal goes into solution in the form of metallic ions bearing a positive charge. The metal therefore becomes negatively charged with reference to the water. The magnitude of the potential difference set up between metal and liquid depends upon the solution pressure of the metal in question. The quantity of metal passing into solution is extremely small, since electrostatic attraction is almost immediately set up between the negatively charged metal and the positive ions in solution, and this prevents further solution of the metal. If the solution already contain a salt of the metal, there is a tendency for the positive metallic ions in solution to separate out on the metal, which therefore becomes positively charged with reference to the solution.

The actual difference of potential between metal and liquid is the resultant of these two opposing forces. While the solution pressure is always constant for one and the same metal, the tendency for the reverse operation to occur varies with the number of metallic ions present in solution. Thus the electric potential of a metallic electrode in contact with a solution of one of its salts is a measure of the concentration of metallic ions in the solution.

When hydrogen is absorbed by finely divided platinum, it behaves in many ways as a metal and exerts a definite solution pressure. Platinum itself has a negligible solution pressure, so that an electrode of platinum coated with platinum black and saturated with hydrogen behaves electrically as if it were metallic hydrogen. The difference of potential set up when such an electrode is dipped into an aqueous solution is therefore a measure of the concentration of hydrogen ions in that solution.

MEASUREMENT OF DIFFERENCE OF POTENTIAL.—It is a difficult matter to measure the absolute difference of potential between a solid electrode and a liquid. This difficulty can be overcome by indirect methods. The method usually adopted is to combine the electrode, through the medium of an intervening vessel containing an appropriate liquid, with a standard electrode of known potential, and to measure the electromotive force of the resulting cell. This standard electrode may consist of platinum saturated with hydrogen and in contact with a solution of known hydrogen ion concentration. In the normal hydrogen electrode, to which all measurements are eventually referred, the solution contains 1 gram. of ionised hydrogen per litre. It contains slightly more hydrochloric acid than the ordinary normal HCl solution, owing to the fact that the dissociation of the acid is not quite complete. Several other solutions of known definite hydrogen ion concentration have been used as standards, but it is more convenient to employ a decinormal calomel electrode. In this the electrode consists of mercury and the liquid is a $\frac{N}{10}$ potassium chloride solution saturated with calomel. The difference of potential between the mercury and solution is 0.613 volt, the mercury being positive and the solution negative.

If this electrode be coupled up with a hydrogen electrode, the current tends to flow outside the cell from the mercury to the platinum, and if the hydrogen electrode be a normal one, the difference of potential between the two electrodes is 0.338 volt. With solutions of lower hydrogen ion concentration the potential difference is, of course, greater than this, since more hydrogen tends to pass from the platinum into the solution, and the platinum becomes more negative with reference to the solution. For some purposes it is preferable to use a calomel electrode made up with $\frac{N}{1}$ potassium chloride, or even a saturated potassium chloride solution. With these electrodes the E.M.F. of the cell has a different value.

RELATIONSHIP OF E.M.F. AND HYDROGEN ION CONCENTRATION.—The following table shows the simple linear relationship which exists between the E.M.F. of the cell when using a $\frac{N}{10}$ calomel electrode, and the strength of the solution in respect of hydrogen ions:

E.M.F. against $\frac{N}{10}$ Calomel Electrode Volts.	Normality of Solution in Respect of Hydrogen Ions.	P_H^+
0.338	$\frac{N}{1}$	0
0.396	$\frac{N}{10}$	1
0.453	$\frac{N}{100}$	2
0.511	$\frac{1.000}{N}$	3
0.569	$\frac{10.000}{N}$	4
0.626	$\frac{100.000}{N}$	5
0.684	$\frac{1.000.000}{N}$	6
0.742	$\frac{10.000.000}{N}$	7
0.800	$\frac{100.000.000}{N}$	8
0.857	$\frac{1.000.000.000}{N}$	9
0.915	$\frac{10.000.000.000}{N}$	10
0.973	$\frac{100.000.000.000}{N}$	11
1.030	$\frac{1.000.000.000.000}{N}$	12

Instead of expressing the hydrogen ion concentration of a solution as, *e.g.*, $\frac{N}{1,000}$, the logarithm of the denominator, termed P_H^+ , is generally used. Thus for a solution of $\frac{N}{1,000}$ strength in respect of hydrogen ions, P_H^+ would be equal to 3, since 3 is the logarithm of 1,000. At the point of absolute neutrality the concentration of hydrogen ions, due to the dissociation of water into $\frac{+}{H}$ and $\frac{-}{OH}$ ions, corresponds to $P_H^+ = 7.12$ at $18^\circ C.$, or $\frac{N}{13,185,000}$, and the E.M.F. against the $\frac{N}{10}$ calomel electrode is 0.748 volt. As we get into the region of alkalinity, the hydrogen ion concentration becomes still smaller—*i.e.*, the value of P_H^+ greater, since, according to the laws of mass action, the product of the concentration of $\frac{+}{H}$ and $\frac{-}{OH}$ ions must be constant. Thus even the most strongly alkaline solutions will show a definite, though extremely minute, concentration of hydrogen ions.

COMPARISON WITH TITRATION METHODS.—The values for acidity obtained by this method are not the same as those given by titration, owing to the varying degree of electrolytic dissociation shown by different acids. Thus a $\frac{N}{10}$ solution of acetic acid gives the same result on titration as $\frac{N}{10}$ hydrochloric acid, since each contains a chemical equivalent of the respective acid in 10 litres. The hydrochloric acid, however, is dissociated to the extent of 91 per cent. into hydrogen and chlorine ions, while only 1.3 per cent. of the acetic acid is in the ionic condition. The hydrogen ion concentration of $\frac{N}{10}$ hydrochloric acid is in the neighbourhood of $P_H^+ = 1$, while that of $\frac{N}{10}$ acetic acid is $P_H^+ = 2.89$, and for $\frac{N}{10}$ solutions of weaker organic acids the hydrogen ion concentration is still lower. A single determination of this value cannot therefore take the place of a titration unless the degree of dissociation of the acid be known, but there are three directions in which the method may be of value in analytical work.

1. The absolute acidity, quite apart from the titration value, may afford an indication of the extent to which a particular substance is likely to act on the metal or other material of the container. The solvent action of various foods upon tin containers and upon lead-glazed earthenware cooking vessels are cases in point.

2. When combined with a titration, the method may give information as to the nature of the acid in solution—that is to say, whether it is a mineral acid or an organic acid, or a mixture of the two. Vinegar adulterated with sulphuric acid would be a case in point.

3. In the titration of dark-coloured solutions, where ordinary indicators cannot be used, portions may be withdrawn at intervals, and successive determinations of the hydrogen ion concentration made until the value $P_H^+ = 7.12$, corresponding to complete neutrality, is reached.

POGGENDORF'S "COMPENSATION" METHOD.—The simplest method of measuring the potential difference is the so-called "compensation" method of Poggendorf, shown diagrammatically in Fig. 1. A is an accumulator, W a thin wire 1 m. in length of 10 to 20 ohms resistance, C the cell of which the E.M.F. is to be measured, S a sliding contact, and E an electrometer or galvanometer connected with a key K. Between the two ends of the wire W there is a constant and regular fall of potential. If we assume that between P and P¹ there is a difference of potential of 2 volts due

to the accumulator current, then at a point X, three-quarters of the way along the wire from P¹, the potential difference between X and P¹ is $\frac{3}{4} \times 2$, or 1.5 volts. If the E.M.F. of the cell C be exactly 1.5 volts, and the sliding contact S be placed at this point, the opposing E.M.F.s. will neutralise each other, and no current will flow through the electrometer E when the key is depressed. Whatever the E.M.F. of the cell C, so long as it is less than 2 volts, a point can be found, by moving the sliding contact S along the wire, at which no current will flow through E. Having found this point, we substitute for C a normal cell C¹ of known E.M.F., and ascertain again the point at which no current flows. If we call these two points X and X¹, then the unknown E.M.F. of the cell is found by simple proportion:

$$C : C^1 = P^1X : P^1X^1.$$

For the most accurate work the resistance wire is replaced by a potentiometer or resistance box, and a delicate galvanometer is used to ascertain the point at which

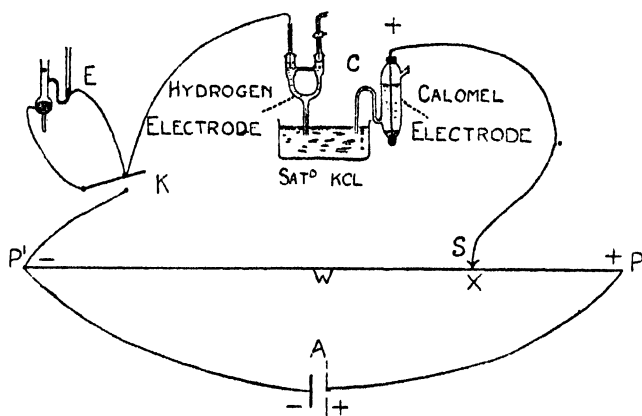


FIG. 1.—DIAGRAM OF POGGENDORF'S COMPENSATION METHOD.

no current flows. The normal cell used for comparison is specially constructed, and is usually calibrated at the National Physical Laboratory. The temperature at which the measurements are made is controlled by immersing the cells in a thermostat.

NORMAL CELL AND ELECTROMETER.—These refinements are not necessary for ordinary work, where an accuracy of 0.002 to 0.003 volt is quite sufficient. A normal cell with poles of cadmium amalgam and mercury showing a constant E.M.F. of 1.0186 to 1.0187 volts at 18° C. is easily made in the laboratory, and will retain its E.M.F. unchanged for many years.

A capillary electrometer, when certain precautions are taken, gives quite satisfactory results, and is easily constructed by any glass-blower. The most important points in preparing this electrometer for use are to get the interior of the capillary perfectly clean by treatment with hot chromic acid, and to use pure mercury and dilute sulphuric acid which has previously been well shaken up with mercury and allowed to stand in contact with the metal for some time. The electrometer is connected with a key (K in Fig. 1) so arranged that its two poles are

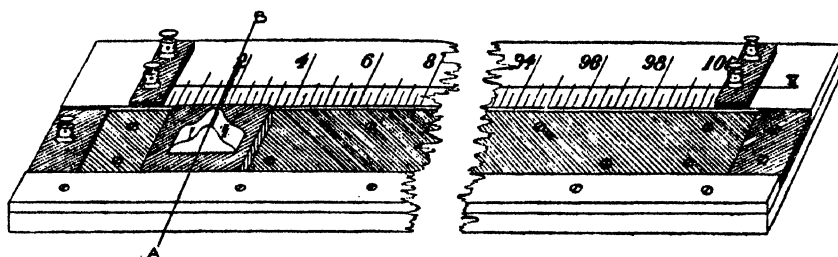


FIG. 2a.—MEASURING BRIDGE.

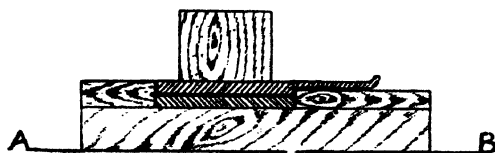


FIG. 2b.—BRIDGE CONSTRUCTION.

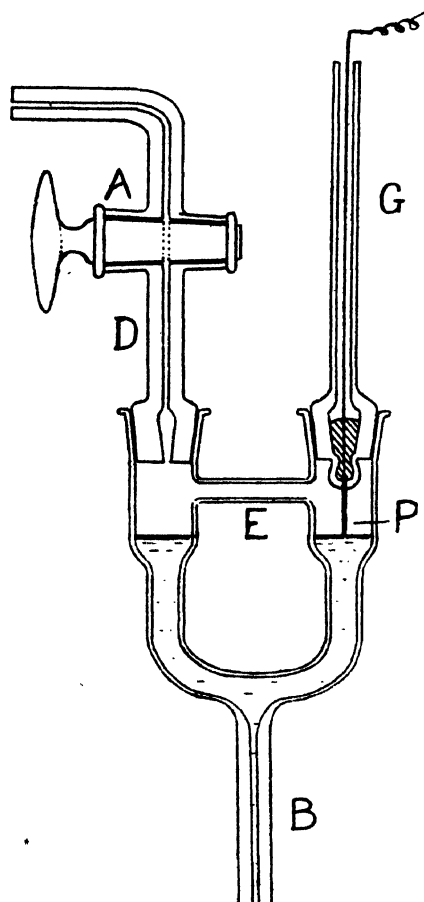


FIG. 3.—HYDROGEN ELECTRODE.

always short-circuited through the key except at the moment when the key is depressed during the taking of a reading.

The $\frac{N}{17}$ calomel electrode is also readily made in the laboratory. It is not possible in a paper of this character to describe the construction of these pieces of apparatus with the necessary completeness of detail. They are fully described and figured in Ostwald's "Physiko-Chem. Messungen," 1902, pp. 333-342, 361-364, 381-383, and later editions. It should be noted that it is electromotive force, and not current, which is measured by this method. While current flows continuously through the accumulator circuit, the electrometer circuit is kept open except during the fraction of a second required for an observation. If this circuit be allowed to remain closed for any length of time, there will be danger of polarisation occurring in the cells and electrometer, leading to irregular results.

RESISTANCE WIRE.—As regards the resistance wire and the hydrogen electrode, I have found the special forms shown in Figs. 2a, 2b, and 3 the most satisfactory.

The former, which was made by Mr. W. A. Godby in my laboratory, consists of a thirty-four gauge "Eureka" wire of approximate resistance 10 ohms per yard, and exactly 1 m. in length, stretched on a board above a metre scale. The two ends of the wire are in contact with brass terminals. The sliding contact is a copper needle passing under the resistance wire and fixed to a metal plate which slides along a brass strip laid the length of the board. Connection between the sliding contact and the electrometer circuit is made by a terminal at one end of the brass strip. The resistance of the brass strip being "nil," this arrangement is equivalent to attaching the wire of the electrometer circuit direct to the sliding contact, and has the advantage that the wire of the circuit does not get into the way when the slide is moved. If desired, the resistance wire can be calibrated by comparison with standard resistances or by Strouhal and Barus' method (Ostwald's "Physiko-Chem. Messungen," 1902, p. 350), but I have not found this necessary, except for very accurate work.

HYDROGEN ELECTRODE.—The construction of the hydrogen electrode is shown in Fig. 3 reduced to approximately three-fifths diameter. The U-tube is closed by two ground-in stoppers, one of which carries a capillary tube D, furnished with a cock A, while through the other passes a wider tube G, with a short piece of stout platinum wire P fused into its lower end. Before use, this wire is coated with platinum black. This is done by immersing the clean wire in 2 to 3 c.c. of a 3 per cent. platinum chloride solution containing a minute trace of lead acetate, and passing the current from an accumulator through the liquid. The wire which is to be coated is made the cathode and another piece of platinum wire the anode. Electrical contact with the fused-in wire is secured by an amalgamated copper rod dipping into a drop of mercury in the bottom of the tube G. After the current has passed for two or three minutes the wire has become sufficiently coated with platinum black. It is then removed from the solution and washed with distilled water. In order to remove the last traces of platinum chloride the electrolysis is repeated for a few minutes, dilute sulphuric acid being used instead of platinum chloride. This has the effect of reducing any residual platinum chloride to platinum. The electrode is then again washed with distilled water and replaced in the U-tube.

To carry out a measurement with this electrode, the U-tube is filled with the liquid to be examined by opening the cock A and drawing the liquid up through the capillary B until both sides of the U-tube are completely filled. Electrolytic hydrogen, of which only a few cubic centimetres are required for each determination, is then passed in through the capillary D, care being taken that no air gets in as well. The hydrogen is allowed to displace the liquid just so far that the tip of the platinum wire P touches the surface of the liquid. This is an important point, as it is found that if the platinum be more fully immersed it is difficult to get constant readings in a short time. The form of the U-tube, with its cross connection E, allows of its being tilted to the required angle for contact to be established without having to adjust the volume of the hydrogen.

When the electrode has been filled it is placed in a holder, and the end of the capillary tube B dipped into a vessel containing saturated potassium chloride. The siphon tube of the calomel electrode is also dipped into the same solution, and the two electrodes connected up as shown in Fig. 1. Care must be taken that the positive and negative poles correspond to those of the accumulator, so that the two currents are opposed in direction.

POTASSIUM CHLORIDE SOLUTION AS CONNECTING LIQUID.—The object of using a saturated solution of potassium chloride as connecting liquid is to avoid errors due to diffusion potential. If two solutions be brought into contact with each other, there is a tendency for ions to migrate or diffuse from one solution to the other across the boundary line. Thus, if a hydrogen chloride solution be brought into contact with a potassium chloride solution, diffusion of hydrogen, chlorine, and potassium ions will occur in both directions in amount proportional to their relative concentration in the two liquids. The migration velocities of the different ions vary considerably, that of hydrogen being much higher than any of the others. Hydrogen ions will therefore diffuse into the salt solution at a greater rate than potassium ions can move in the reverse direction, and the salt solution will become positively charged with reference to the HCl. The potential difference thus set up will, unless allowed for, cause an error in the reading when the circuit is closed. If the two solutions be separated by a highly concentrated solution of potassium chloride in which the migration velocities of anion and kation are approximately the same, the diffusion of hydrogen, relatively to that of the other ions present, is reduced to a negligible amount, and the error from this source is avoided.

The cell of which the E.M.F. is to be measured is therefore the following: (Pt.H₂: solution: saturated KCl: ($\frac{N}{10}$ KCl: Hg₂Cl₂: Hg).

When the point on the resistance wire has been found at which no current passes on depressing the key of the electrometer, the connections are changed over to the standard cell and another reading taken. The E.M.F. of the first cell is found by simple proportion, and the hydrogen ion concentration of the solution can be ascertained by interpolation from the table given above.

ACCURACY OF THE METHOD.—In comparing the accuracy of the method with ordinary titration we may take the case of $\frac{N}{10}$ hydrochloric acid. In a titration one will take, say, 25 c.c. of the liquid, and titrate with $\frac{N}{10}$ sodium hydroxide solution, with an accuracy of 0.1 c.c., or 1 in 250. In the electrical method a $\frac{N}{10}$ hydrochloric

acid solution, of which only 5 to 10 c.c. are required, gives an E.M.F. of 0.398 volt when coupled up with a $\frac{N}{10}$ calomel electrode. The E.M.F. at absolute neutrality is 0.748 volt, the difference between the two being thus 0.350 volt. If the potential difference between the two ends of the resistance wire be reduced to, say, 1.5 volts by the insertion of a small coil of thin wire in the accumulator circuit, 0.350 volt will correspond to a length of $\frac{0.3}{1.5} \times 50$ m. on the wire, or 233 mm. With the apparatus described it is possible to read to 0.5 mm., but in ordinary work the error, unless the wire is calibrated and the temperature of the electrodes carefully regulated, will usually amount to 0.002 to 0.004 volt, corresponding to 1.5 to 3 mm. on the wire. This represents a degree of accuracy of 1 in 100 to 200, as compared with 1 in 250 by titration.

It is more particularly with weak organic acids that the method is of the greatest value. Such solutions can often not be titrated satisfactorily. The colour change of the indicator is too gradual, and the type of indicator employed has a great influence on the result. The electrometric method gives us exact values for acidity or alkalinity comparable with each other, whatever the nature of the solutions examined.

CONTROL TEST.—The accuracy of the measurements can be controlled by a test carried out on a solution of known hydrogen ion concentration. A convenient solution for this purpose is: 10 c.c. *N* sodium hydroxide solution; 20 c.c. *N* acetic acid; and 70 c.c. distilled water.

If this solution be introduced into the hydrogen electrode vessel and coupled with a $\frac{N}{10}$ calomel electrode, the E.M.F. of the cell should be 0.6045 volt at 18° C. Several other such solutions are given by G. S. Walpole (*Biochem. J.*, 1914, 8, 628-640). Walpole has also described a simple arrangement for introducing hydrogen into the electrode which is particularly convenient for electrometric titration, and has prepared a useful chart giving the more important data for different standard solutions and indicators (*Biochem. J.*, 1913, 7, 410; 1914, 8, 131).

LIQUIDS CONTAINING DISSOLVED GASES.—Liquids containing gases in solution require special consideration, according as the gas is active in an electromotive sense or not. To the former class belong free chlorine and free ammonia. If these be present in the liquid and liable to be given off into the hydrogen atmosphere, the method cannot be used. The platinum will behave partly as a hydrogen electrode and partly as a Cl or NH_4 electrode, and the potential difference will be dependent not only upon the concentration of H-ions, but also upon that of Cl or NH_4 ions in the liquid. Carbonic acid gas is not active in the same way, but errors may be produced by the escape of carbon dioxide from the liquid into the hydrogen atmosphere. The surface layers of the liquid thus become gradually more alkaline, owing to escape of carbon dioxide, and the reading will not become constant until equilibrium is established. This question is fully discussed by Walpole (*Biochem. J.*, 1913, 7, 424-426). With the U-shaped electrode the manipulations necessary to obtain gaseous equilibrium between the solution and the hydrogen are easily carried out. The error due to the reduction in the partial pressure of the hydrogen by the presence of carbon dioxide is small, and may be neglected.

DETAILS AND PRECAUTIONS.—A few practical points about the apparatus may be mentioned. All connecting wires should be well insulated, and constructed of stout

copper or other metal with high conductivity, so as to avoid the introduction of resistance except where it is wanted. For the same reason all contact points must be kept clean and firmly screwed up. Care must be taken that nothing in the nature of a short circuit occurs through drops of water on the bench or on the resistance board. The changing over from the hydrogen cell to the standard cell is facilitated by a simple switchboard with holes containing mercury, into which the ends of the connecting wires are dipped. The ends of the wires must be well amalgamated to ensure good contact. Small potential differences may be measured in series with the standard cell, and a second reading taken with the two cells in opposition. The difference between the two readings, divided by two, gives the reading due to the experimental cell alone.

The actual cost of the apparatus, apart from the accumulator, is not great. It will naturally depend to a great extent on the technical resources of the laboratory, but in most cases £2 to £3 should cover it all. Where facilities exist for glass-blowing and for simple wood and metal working it could be made for much less. The inch or two of platinum wire required for the hydrogen electrode and for the contacts of the standard cell, the electrometer, and the calomel electrode will cost at most a few shillings.

APPLICATION OF THE METHOD.—As an instance of the application of this method to analytical problems, the results given below are of interest. They were obtained during an investigation of the action of various foodstuffs when cooked under practical conditions in lead-glazed earthenware casseroles (*cf.* H. Masters, *ANALYST*, 1919, 44, 164).

Nature of Food Cooked.	E.M.F. against $\frac{N}{10}$ Calomel Electrode.	H-Ion Concentration P_H^+ .	Ratio of Lead dissolved per Unit of Surface to that dissolved subsequently from same Vessel by Heating to approximately 95° C. for Two Hours with 1 per cent. Citric Acid.
	Volts.		
Rice, tomato, and onion...	0.649	5.40	0.064
Beans, cabbage, potato, turnip, and onion ...	0.647	5.36	0.043
Steak, kidney, and onion...	0.676	5.86	0.217
Rhubarb	0.495	2.73	0.209
One per cent. citric acid...	0.467	2.22	1.0

LIMITATIONS OF THE METHOD.—The course of a typical electrometric titration is shown in Fig. 4, which represents the titration of a dilute solution of hydrochloric acid with $\frac{N}{10}$ sodium hydroxide solution. The point of absolute neutrality is that at which the curve is steepest—*i.e.*, at which the hydrogen ion concentration changes most rapidly with successive additions of sodium hydroxide. In this particular case neutrality was reached with 11.3 c.c. of $\frac{N}{10}$ sodium hydroxide solution. It can be seen from this figure that in strongly acid or alkaline solutions

the change in hydrogen ion concentration is relatively small for a given change in the "titration acidity" of a solution. As we approach the neutrality point, the rate of change in hydrogen ion concentration becomes relatively much greater. The method is therefore not well adapted to the measurement of high acidities, and its main value lies in its power of differentiating between slight variations in reaction in the neighbourhood of the neutrality point. The subject of electrometric

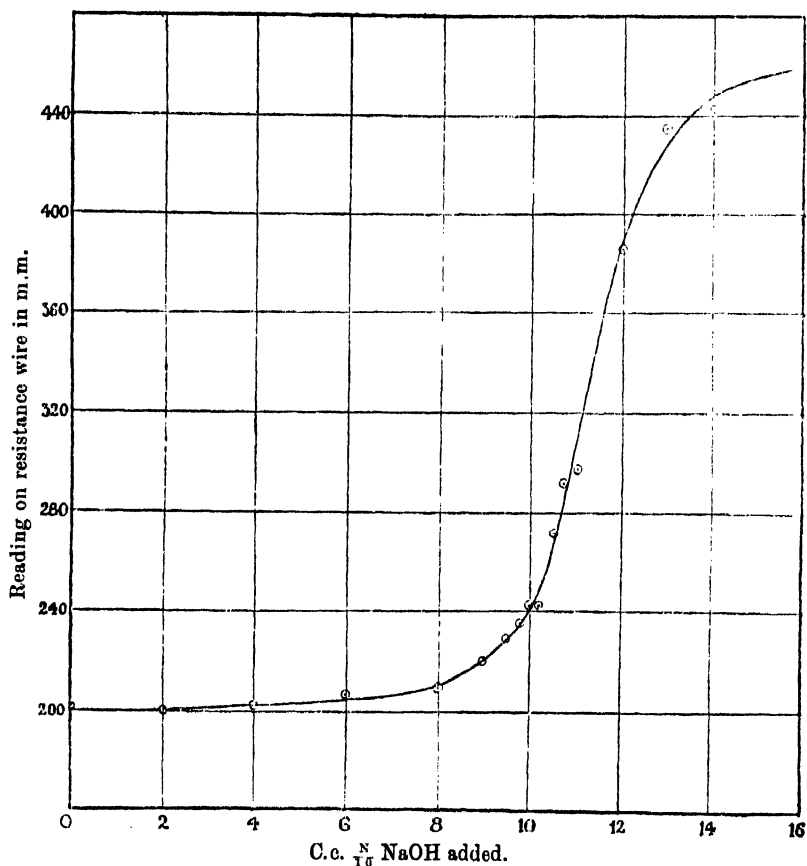


FIG. 4.—TYPICAL ELECTROMETRIC TITRATION.

titration as applied more especially to tan liquors was investigated by H. J. S. Sand, D. J. Law, and J. T. Wood (ANALYST, 1911, 36, 121, 515), who described a special form of hydrogen electrode for this purpose.

My thanks are due to my assistant, Mr. W. A. Godby, for preparing the drawings given in this paper.

A FURTHER CONTRIBUTION TO THE STUDY OF SOUTH AMERICAN OILSEEDS.

By G. T. BRAY, A.I.C., AND H. T. ISLIP, A.I.C.

DURING recent years considerable attention has been given to South America as a possible source of commercial supplies of oilseeds yielding oils and fats of technical importance. A large number of oilseeds from that country have been examined from time to time at the Imperial Institute, and the results of some of these investigations have already been published (*cf. Bull. Imp. Inst.*, 1917, 15, 479; 1919, 17, 186; 1920, 18, 168, 172; Bray and Elliott, *ANALYST*, 1916, 41, 298), whilst papers on this same subject by other authors have also appeared in the *ANALYST* (Bolton and Hewer, 1917, 42, 35; Bolton and Revis, 1918, 43, 251).

In this paper an account is given of the results of examination of four further varieties of South American oilseeds.

"CUPU" SEEDS.—These seeds are derived from *Theobroma grandiflorum* (N.O. *Sterculiaceæ*), a tree which occurs in the district of the Upper Amazon and in the province of Para. The seeds are flat and roughly ovate, with a slight odour resembling that of cocoa. They consist of a thin, brittle, closely adhering, reddish-brown husk, covering a moderately soft, pale brown, oleaginous interior. The average dimensions of the seeds received at the Imperial Institute were 1.0 inch long, 0.8 inch broad and 0.25 inch thick, whilst the average weight of a seed was 2.2 grms.

The fat extracted with petroleum spirit was creamy white, of rather soft consistence, and practically devoid of taste or odour. From the chemical examination it would appear that this fat resembles cacao butter. Cupu fat, however, is softer than cacao butter, and lacks its brittleness and odour. It could probably be used for edible purposes.

The residual meal contained 1.0 per cent. of an alkaloidal body which gave the murexide reaction and was probably theobromine. This amount of alkaloid corresponds to 0.5 per cent. on the original seeds.

HYMENÆA FRUITS.—These fruits are derived from a species of *Hymenæa* probably *H. Courbaril* (N.O. *Leguminosæ*), which is found in South America and in Jamaica. The sample examined at the Imperial Institute consisted of large brown, rough, shiny pods about 5.7 inches long, 2.3 inches broad, and 1.2 inches thick. The pod-cases were about 0.2 inch thick, and were very hard and brittle. The pods contained on an average seven seeds embedded, generally together with a few small lumps of pale yellow resin, in a pale reddish-brown, soft, mealy material, which clung tenaciously to them. The seeds were of flattened oval shape, and had a very hard, tough, smooth, chocolate-coloured seed coat. The kernels were very hard, and of a pale cream colour internally. The average weight of a seed was 3 grms. The pods consisted of: Pod-cases, 74 per cent.; mealy material and resin, 6 per cent.; and seeds, 20 per cent.

The oil extracted from the seeds with petroleum spirit was nearly colourless, and had rather an unpleasant odour. Owing to the small amount of material available, the constants of the oil were not determined.

The residual meal was pinkish-brown and possessed a pleasant agreeable taste, but was found to be rather poor in protein. The mealy substance surrounding the seed is palatable, and is stated to be eaten by the natives of the West Indies.

PARINARIUM SEEDS.—The fruits of several species of *Parinarium* are already known to be oleaginous. The seeds of *P. Mobola* have been imported as an oilseed into Europe under the name of "Mabo" seeds, whilst the oil from *P. senegalense* seeds is occasionally used in Senegal for soap-making.

The present sample consisted of egg-shaped fruits with a reddish-brown, rough surface, marked by small irregular cracks. The dimensions of the fruits were from 2 to 3 inches long, and from 1.4 to 2.0 inches in diameter at the widest part. The shell, from 0.3 to 0.45 inch thick, was very tough and fibrous, and not easily removed from the kernel. The kernels were yellow and oily, but of firm consistence, and split easily, disclosing a long central cavity. They were covered with a light greyish-brown inner fibrous skin and a reddish-brown outer skin, both of which were readily detachable. The odour of the kernels was characteristic and unpleasant. The fruits were composed of 79 per cent. of shell and 21 per cent. of kernels. The average weight of a fruit was 29.3 grms. and of a kernel 7.6 grms.

The oil extracted with petroleum spirit was a thick, viscid, dark-brown oil, which on keeping became a soft semi-solid fat. It had a strong unpleasant odour. This oil differs considerably from a sample of "Po-yoak" oil from Sierra Leone examined at the Imperial Institute (*Bull. Imp. Inst.*, 1918, 16, 38), which is probably derived from the nuts of another species of *Parinarium*. The latter had drying properties, and polymerised to a solid mass on heating to 300° C. for twenty minutes in an oil-bath. The "Po-yoak" seeds are more globular and have a thinner shell than this South American variety.

The residual meal had an unpleasant bitter taste.

PLATONIA SEEDS.—These seeds were derived from a species of *Platonia* (N.O. *Guttiferae*). They were somewhat irregular in shape, but their predominating form was a flattened ovoid, slightly concave on one side, their average dimensions being 2 inches long, 1 inch broad and $\frac{3}{4}$ inch thick. These seeds had a thin, closely adhering, tough brown skin, enclosing a firm oleaginous kernel, which varied in colour from greyish-white to brown, but in many seeds was brown throughout. The flesh in places showed minute cavities containing brown resinous material. The average weight of a seed was 11.8 grms.

The oil extracted with petroleum spirit was a dark-brown solid fat of fairly firm consistence, and having a slight aromatic odour.

The residue from the extraction with petroleum spirit yielded, on extraction with acetone, 3 or 4 per cent. of a soft brown resinous material. The residual meal was free from alkaloids and cyanogenetic glucosides.

The following table gives the results obtained in the examination of these seeds and the oils extracted from them :

	<i>Theobroma grandi- florum</i> Seeds.	<i>Hymenaea Courbaril</i> Seeds.	<i>Parinarium</i> Species Kernels.		<i>Platonia</i> Species Seeds.
			South America.	Sierra Leone.	
<i>Composition :</i>					
Moisture, per cent.	8.2	11.5	3.4	8.7	3.2
Oil on material as received, per cent.	48.7	6.4	74.2	58.3	75.0
Oil on material dried at 100° C., per cent.	53.0	7.2	76.8	63.8	77.5
<i>Analytical Values of Oil :</i>					
Melting-point, ° C.*	32.0	—	—	—	31.0
"Titre" of fatty acids, ° C.	48.1	—	41.6	48.3	50.1
Specific gravity	0.8522†	—	0.905†	0.969†	0.8782†
Acid value	44.0	—	16.2	17.4	46.4
Saponification value	187.8	—	200.5	192.3	199.5
Iodine value (Hübl, 17 hours), per cent.	44.8	—	77.3	157.1	77.8
Unsataponifiable matter, per cent. ...	0.91	—	0.76	0.7	3.63
Volatile acids, soluble§	0.08	—	2.68	0.2	0.13
Volatile acids, insoluble§	0.12	—	0.52	0.4	0.37
Refractive index [<i>n</i>] _D ⁴⁰	1.456	—	1.469	—	1.469
<i>Composition of Residual Meal :</i>					
Moisture, per cent.	9.9	11.0	7.4	12.2	9.2
Crude proteins, per cent.	18.7	7.1	24.7	12.1	14.3
Fat, per cent.	7.0	7.0	7.0	7.0	7.0
Carbohydrates, etc. (by difference), per cent.	43.8	67.7	46.6	56.1	46.2
Crude fibre, per cent.	14.3	5.5	8.2	8.9	13.4
Ash, per cent.	6.3	1.7	6.1	3.7	9.9
<i>Nutrient ratio</i>					
Nutrient ratio	1 : 3.2	1 : 11.8	1 : 2.5	1 : 6.0	1 : 4.4
Food units	108	103	126	104	99

* Open tube method.

† At 100°/15° C.

‡ At 15°/15° C.

§ C.c. of $\frac{N}{10}$ potassium hydroxide solution required to neutralise the volatile acids from 5 grms. of the oil.

CONCLUSIONS.—Of the four oilseeds described, "Cupu" (*Theobroma grandiflorum*) is the only one which can be considered as at all promising from a commercial point of view. The quantity of these seeds available is not known, so that it is not possible to state whether they can be exported in sufficiently large consignments to be of economic value.

The authors desire to express their thanks to Mr. T. B. Woodward, of Liverpool, for kindly supplying the seeds for examination.

NOTES.

The Editor desires to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

A SAMPLE marked "Chilian honey" was submitted for my opinion as to the risk of its sale as honey under the Food and Drugs Acts, even with the reservation of the word "Chilian."

The analysis as made by my chief assistant, Dr. H. E. Cox, F.I.C., M.Sc., was: Water, 21.50; ash, 2.0; cane sugar, 16.50; glucose, 4.0; raffinose, dextrin, proteins, traces; and invert sugar, 56.0. Total, 100.0.

In view of genuine honey giving, say, 75 per cent. of invert sugar with 20 per cent. of water, and about 5 per cent. cane sugar, dextrin, etc., I gave the opinion that the excess of cane sugar alone would preclude the sample being considered pure and unsophisticated honey.

What exactly "Chilian honey" may be is unknown to me; but if the above was genuine, the habits of the bees of Chili might repay investigation!

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REPORTS OF PUBLIC ANALYSTS.

The Editor would be glad to receive the Annual or other Reports of Public Analysts containing matter of interest to the Society. Notes made from such Reports would be submitted to the Publication Committee.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Mineral Constituents of Potatoes and Potato Flour. C. E. Mangels. (*J. Ind. Eng. Chem.*, 1921, 13, 418-419.)—Four samples of potatoes from different sources yielded the following results: Total ash, 4.31 to 5.70; calcium oxide, trace to 0.161; magnesium oxide, 0.236 to 0.290; potassium oxide, 2.12 to 2.69; phosphoric anhydride, 0.685 to 0.730; sulphur, 0.108 to 0.164; and chlorine, 0.076 to 0.260 per cent. The flours prepared from these potatoes contained: Total ash, 3.51 to 4.90; calcium oxide, trace to 0.145; magnesium oxide, 0.198 to 0.279; potassium oxide, 1.82 to 2.51; phosphoric anhydride, 0.438 to 0.655; sulphur, 0.105 to 0.155; and chlorine, 0.066 to 0.197 per cent. All these results are expressed as percentages of the dry substance. The relative distribution of the constituents of the ash is not altered appreciably during the process of manufacture of the flour; bolted flour made

from peeled potatoes has practically the same composition as unbolted flour made from unpeeled potatoes, but the former has a better colour. W. P. S.

Carbohydrate Content of the Navy Bean and Peas. W. H. Peterson and H. Churchill. (*J. Amer. Chem. Soc.*, 1921, **43**, 1180-1185.)—Analyses made by the methods of the Association of Official Agricultural Chemists of navy beans, Alaska garden peas, and Canada field peas, showed the nitrogen-free extracts to amount to 58.97—61.80, 60.26 and 54.47 per cent., whilst the starch present was 35.20—50.54, 51.21, and 45.11 per cent. respectively. Extremely fine grinding of the material is essential for the complete digestion of the starch by diastase solution, an ordinary feed-mill grinding yielding from 10 to 12 per cent. less starch. The blue colour given on the addition of iodine is too sensitive to indicate when the whole of the starch is saccharified, and other substances present may also show this reaction. A digestion for four hours with malt extract is probably sufficient to convert the starch completely enough for analytical purposes. Tables showing the kind and amount of carbohydrates contained in the various extracts, the complete analysis of navy beans, and the effect of varying digestion periods upon the estimation of starch by diastase are given. T. J. W.

Structure of Foreign Haricot Beans. N. T. Giung. (*Comptes rend.*, 1921, **172**, 1436-1438.)—The small haricots cultivated in tropical countries are usually grouped under the name of *Phaseolus Mungo* (L.), although they include three distinct species, *P. Mungo*, *P. radiatus* (Roxb.), and *P. aureus* (Roxb.). The various species of *Phaseolus* may be differentiated by the structure in their seminal teguments, which, in the case of *P. Mungo* and *P. aureus*, are composed of three layers, as compared with five in the case of *P. vulgaris* and *P. multiflorus*. On the other hand, the teguments of certain other species such as *P. calcaratus* have also three layers, but show a very different sub-epidermic structure. The tegument of *P. Mungo* has (1) an epidermic layer, composed of cells the cavity of which contains a green substance, and is club-shaped; (2) a sub-epidermic layer, the cells of which (also containing a green substance) are shaped like dumb-bells, and show regular hexagonal openings between them; and (3) a parenchyme composed of cells with thin walls ranged tangentially, those of the last layers being very flattened. In the case of *P. aureus* the cells of the sub-epidermic layer are less regular in form, and the passages between the cells are irregularly oval, instead of hexagonal, and are also less constantly present. As a rule, the seeds of *P. Mungo* have a blackish-green tegument, more or less covered with yellow-orange spots, and have a somewhat prominent hilum, whilst those of *P. aureus* have a more uniformly green tegument and a hilum which is, at most, only slightly prominent. The green haricot of Indo-China agrees in morphological characteristics with *P. aureus*.

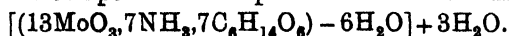
Amylases of the Cereal Grains—Rye. J. L. Baker and H. F. E. Hulton. (*J. Chem. Soc.*, 1921, **119**, 805-809.)—Potato starch paste is rapidly liquefied by ungerminated rye diastase at 50° C., and the conversion products are easily separated by precipitation with alcohol. About 22 per cent. of the original starch is obtained

as a dextrin, having the constants $R_{500} 1.5$ and $[\alpha]_{D^{20}} = 184.1^\circ$, the only other product yielded being maltose, which was readily crystallised out in a state of purity after evaporation of the alcoholic solution. The dextrin, on degradation for twelve hours at 50°C . with rye amylase, and also with malt amylase, yielded products of similar character to those obtained by the enzymes of ungerminated and germinated barley upon α -amylodextrin previously described by one of the authors (*J. Chem. Soc.*, 1902, **77**, 1177). Repeated precipitation by alcohol of the dextrin produced by rye amylase caused no change in the rotatory or reducing powers, and this substance is, therefore, probably identical with the previously described α -amylodextrin. The amylase of germinated rye, under the above conditions, yields crystalline maltose and an unfermentable reducing dextrin having $R = 10.8$ and $[\alpha]_D = 181.9^\circ$. No intermediate malto-dextrins are produced, and practically the whole of the apparent maltose is fermentable by yeast. T. J. W.

Influence of Ammonium Molybdate on the Rotatory Powers of Certain Sugars. G. Tanret. (*Comptes rend.*, 1921, **172**, 1363-1365.)—As previous authors have observed, the presence of ammonium molybdate produces a considerable change in the specific rotation of pentoses and hexoses, whereas hydrolysable sugars and cyclic compounds such as quercitol and *L*-inositol are not affected in this way. It is suggested that the effect produced by the molybdate may be utilised as a means for estimating certain sugars by isolating the complexes in crystallisable form.

T. H. P.

Influence of Ammonium Molybdate on the Rotatory Power of Mannitol. G. Tanret. (*Comptes rend.*, 1921, **172**, 1500-1503.)—The slight lævo-rotation of mannitol is changed in sign by addition to the solution of ammonium heptamolybdate, the dextro-rotation reaching a maximum when the relationship between the amounts of the two compounds corresponds with the formula:



A well-defined complex compound of this composition is readily isolated by simple crystallisation.

T. H. P.

Table for the Estimation of Dextrose, Invert Sugar, and Lævulose by the Thiocyanate-Potassium Iodide Method. G. Bruhns. (*Chem. Zeit.*, 1921, **45**, 486-487.)—The method is as follows: Ten c.c. of copper sulphate solution (69.5 grms. per litre), 10 c.c. of alkaline tartrate solution (346 grms. of Rochelle salt and 100 grms. of sodium hydroxide per litre), 10 c.c. of sugar solution (containing not more than 0.9 per cent. of sugar), and 10 c.c. of water are mixed, heated to boiling, and boiled for exactly two minutes; 50 c.c. of cold water are then added, the mixture is cooled rapidly, 5 c.c. of potassium thiocyanate-iodide solution (65 grms. of potassium thiocyanate and 10 grms. of potassium iodide per 500 c.c.) and 10 c.c. of 6*N* hydrochloric acid are introduced, and the mixture is at once titrated with thiosulphate solution (34.4 grms. of the salt and 2 c.c. of *N*/1 sodium hydroxide solution per litre; the strength of this solution is 0.1387*N*), starch solution being added as indicator towards the end of the titration. A similar mixture, containing the same quantities of

the reagents without the sugar solution, is titrated at the same time. The difference between the two titrations, in terms of c.c. of thiosulphate solution, is a measure of the sugar present, the quantity of the latter being found by reference to a table. The following values are taken from the full table given in the original:

Thiosulphate Solution.	Dextrose.	Invert Sugar.	Lævulose.
C.c.	Mgrms.	Mgrms.	Mgrms.
0·1	0·45	0·45	0·45
0·5	2·20	2·30	2·35
1·0	4·40	4·60	4·70
1·5	6·60	6·90	7·10
2·0	8·80	9·20	9·45
2·5	11·05	11·50	11·95
3·0	13·25	13·80	14·40
4·0	17·65	18·45	19·25
5·0	22·15	23·15	24·10
6·0	26·70	27·85	29·05
7·0	31·30	32·65	34·05
8·0	35·95	37·45	39·10
9·0	40·70	42·35	44·20
10·0	45·50	47·35	49·30
11·0	50·35	52·35	54·50
12·0	55·30	57·45	59·75
13·0	60·30	62·00	65·05
14·0	65·40	67·85	70·40
15·0	70·60	73·15	75·80
16·0	75·85	78·55	81·30
17·0	81·20	84·00	86·85
18·0	86·70	89·53	92·45
19·0	92·25	95·15	98·10

W. P. S.

Composition of Grape-Seed Oil. E. André. (*Comptes rend.*, 1921, 172, 1413-1415.)—The high figures recorded for the acetyl value of grape-seed oil (13·3 to 144·5) have been attributed, though without sufficient proof, to the presence of ricinoleic acid. Hydroxy fatty acids could not be isolated from a sample of the oil by any of the usual methods of analysis, these only indicating the presence of palmitic, stearic, oleic, and linolic acids, but by fractional crystallisation of the lithium salts from dilute alcohol the hydroxy acids were separated from the solid and liquid acids. The mixed fatty acids (freed from unsaponifiable matter) from 125 grms. of grape-seed oil (acetyl value, 49·3) were dissolved in 3 litres of 70 per cent. alcohol, and the solution boiled for an hour with 40 grms. of lithium carbonate beneath a reflux condenser, and then left in a cold place for twenty-four hours. The first crystals consisted mainly of the lithium salts of solid fatty acids (iodine value, 32); the solution was concentrated by distillation, and then yielded crystals of lithium salts of the liquid fatty acids (iodine value, 131); whilst the mother liquor contained the lithium salts of the hydroxy fatty acids. When isolated, these

were of viscous consistence, and had iodine value 110 and molecular equivalent 277. The average yields from the mixed fatty acids were: Solid acids, 12.5; liquid acids, 62.5; and viscous acids, 25.0 per cent. The low molecular equivalent of the hydroxy fatty acids did not support the view that they consisted of ricinoleic acid.

Estimation of Glycerol in Wine by Conversion into Acrolein. A. Heiduschka and F. Englert. (*Zeitsch. anal. Chem.*, 1921, **60**, 161-166.)—A method is described for estimating glycerol in wine based on its conversion into acrolein by boric acid, as suggested by Wohl and Neuberg (*Ber.*, 1899, **32**, 1352). The extract from 100 c.c. of wine is mixed with 15 c.c. of water, and evaporated to 2 to 3 c.c. in a quartz retort heated on a bath of melted alloy. One gram. of boric acid is added, and the mixture heated until evolution of steam ceases. The retort is then connected with two Péligré tubes immersed in a freezing mixture and containing 20 c.c. of 25 per cent. ammonia; a tube from an air reservoir is passed through the tubulure of the retort. The bath is gradually heated to 320° C., formation of acrolein commencing at 250° C.; a current of air is then slowly passed through the vessel, and heating is continued for forty-five minutes. The contents of the Péligré tubes are then treated with 5 c.c. of $\frac{N}{10}$ silver nitrate solution and warmed until a faint ammoniacal reaction persists. The precipitated silver is filtered off through glass-wool, the filtrate is treated with 5 c.c. of saturated ferric ammonium sulphate solution and dilute nitric acid until decolorised, and the silver is estimated with $\frac{N}{10}$ ammonium thiocyanate. The amount of glycerol may then be found by the aid of a table giving the amount of silver nitrate reduced by pure glycerol. The results by this method are lower than those obtained by the lime and iodide methods and are more nearly in agreement with the theoretical. They are not affected by any substances present in wine, or by other aldehydes, such as croton aldehyde, formed by the action of boric acid.

W. J. W.

Polarimetric Estimation of Tannin in Hops. A. R. Ling and D. R. Nanji. (*J. Inst. Br.*, 1921, **27**, 310-313.)—The following method is based upon previous work by Chapman (*ANALYST*, 1907, **13**, 646), which indicated that 1 gram. of cinchonine is precipitated by 1.217 grms. of hop tannin. Ten grms. of hops are weighed out and extracted with water for two hours on a boiling water bath. After cooling, the mixture is diluted to 508 c.c., filtered, and 100 c.c. of the filtrate is evaporated to slightly less than half its volume, and 50 c.c. of standard cinchonine sulphate (approximately 1 per cent.) are added. The amount of cinchonine present in the clear solution is estimated by the optical rotation, using Landolt's figure of $[\alpha]_D$ 170.3°, and a similar reading is taken of the original cinchonine solution. From these readings, allowing for the dilution of the alkaloidal solution by the hop extract, the amount of cinchonine precipitated is calculated from which the quantity of hop tannin present is deduced. The results obtained are in good agreement with those given by Chapman's gravimetric method.

T. J. W.

The Turner Reaction for Gurjun Balsam. J. B. Luther. (*J. Assoc. Off. Agric. Chem.*, 1921, **4**, 422-424.)—The method described in the U.S. Pharmacopœia

for the detection of gurjun balsam in copaiba (and known as the Turner reaction) has the disadvantage that the concentrated sulphuric acid is liable to char the mixture at the point of contact, and so obscure the coloration. This defect is eliminated by modifying the test as follows: Four drops of the oil are dissolved in 1 c.c. of glacial acetic acid, 1 drop of 10 per cent. sodium nitrite solution is added, and 2 c.c. of a 5 per cent. (by vol.) sulphuric acid solution in glacial acetic acid are then introduced and mixed quickly. A violet coloration indicates the presence of gurjun balsam. Colorations which develop after the lapse of ten seconds must be ignored.

W. P. S.

New Lead Number Estimation in Vanilla Extracts. H. J. Wichmann. (*J. Ind. Eng. Chem.*, 1921, **13**, 414-418.)—In the ordinary method of estimating the lead value of vanilla extracts the precipitation is not complete, being only that which occurs under an arbitrary set of conditions. To obtain a maximum precipitation and to save time, a method is described in which estimations of the lead value and alcohol content are combined. A mixture of 175 c.c. of water, 25 c.c. of 8 per cent. neutral lead acetate solution, and 50 c.c. of vanilla extract is distilled; 200 c.c. of distillate are collected, and the specific gravity of this distillate gives the alcohol content. The residue in the distillation flask is transferred to a 100 c.c. flask with water free from carbon dioxide, cooled, diluted to 100 c.c., and filtered. Ten c.c. of the filtrate are treated with 25 c.c. of water, 10 c.c. of dilute sulphuric acid, and 100 c.c. of 95 per cent. alcohol; the lead sulphate is collected, washed with alcohol, dried, ignited, and weighed. A control estimation is carried out at the same time, using water containing 5 drops of glacial acetic acid in place of the vanilla extract, and the number of grms. of lead precipitated by 100 c.c. of the extract is then calculated. The results obtained are about 34 per cent. higher than those found by the usual method; the lowest value obtained for an undiluted genuine vanilla extract was 0.55. The presence of sugar, glycerol, and coumarin does not affect the accuracy of the method, but the method must be modified as follows if added vanillin is present in the extract: Fifty c.c. of the extract are shaken with 25 c.c. of ether; 25 c.c. of petroleum spirit are then added, and the mixture is again shaken; if, after standing, the mixture does not separate into a brown aqueous layer and a colourless ethereal layer, further small quantities of petroleum spirit are added. The aqueous layer is drawn off, and the ethereal solution washed with 2 c.c. of water. The extraction with ether and petroleum spirit is repeated, and the aqueous solution then used for the estimation of the lead value.

W. P. S.

Essential Oil from Leaves of *Skimmia laureola*. J. L. Simonsen. (*J. Soc. Chem. Ind.*, 1921, **40**, 126-127.)—By distillation in steam at 25 lbs. pressure of the leaves of *Skimmia laureola*, 0.5 per cent. of an emerald green oil was obtained: Sp. gr. (30/30°), 0.9041; $[\eta]_D^{20}$, 1.4648; acid value, 8.63; saponification value, 197.96; saponification value after acetylation, 238.6. By prolonged fractional distillation of this oil under a pressure of 200 mm., the following fractions were isolated: (1) 130-140°, 0.46 per cent.; (2) 140-145°, 4.6 per cent.; (3) 145-150°, 0.61 per cent.;

(4) 150-155°, 8.4 per cent.; (5) 155-160°, 2.3 per cent.; (6) 160-167°, 2 per cent.; (7) 167-173°, 50 per cent.; (8) 173-177°, 0.61 per cent.; (9) 177-182°, 1.05 per cent.; (10) 182-190°, 1.2 per cent.; and (11) above 190°, 18.2 per cent. From fractions 1, 2, and 3, on redistillation, a considerable fraction was obtained which boiled at 130-135° (200 mm.) and at 176-182° (695 mm.). Analysis showed: C, 85.5; and H, 11.6 per cent. Its constants were: Sp. gr. (30/30°), 0.859; $[\eta]_{D}^{20}$, 1.471; and $[\alpha]_{D}^{20}$, -4.11. The nature of the hydrocarbon of which these fractions apparently consist was not definitely determined. Fraction 4 consisted of nearly pure *l*-linalool, and fractions 5 and 6 were mixtures of *l*-linalool and *l*-linalyl acetate. The bulk of the oil which was obtained in fraction 7 consisted of pure *l*-linalyl acetate; its constants were: Sp. gr. (30/30°), 0.892; $[\eta]_{D}^{20}$, 1.4537; and $[\alpha]_{D}^{20}$, -7.25°. Fractions 8 and 9 consisted chiefly of linalyl acetate; the latter showed traces of a high-boiling alcohol after hydrolysis; this was also the case with fraction 10. No fraction of constant b.-pt. could be obtained from fraction 11. The carbon content of the various fractions varied from 76.1 to 81.0 per cent., and the hydrogen from 10.3 to 11.2 per cent. Those fractions distilling above 175° varied in colour from emerald green to indigo blue, and the higher boiling fractions showed a blue fluorescence, but methyl anthranilate was absent.

W. J. W.

Volatilisation of Ethyl Nitrite from Sweet Spirit of Nitre. J. G. Roberts.

(*Amer. J. Pharm.*, 1921, **93**, 320-324.)—Experiments are described designed to determine the influence of the various factors concerned in the storage and dispensing of sweet spirit of nitre, which lead to the loss of ethyl nitrite. The results obtained clearly show that the chief loss is due to evaporation, which may arise from agitation and exposure to air, the frequent sampling from a large bottle, and the storage of small amounts of the drug in large bottles. Direct sunlight also causes a rapid decomposition of the ethyl nitrite when small white flint glass bottles are completely filled with spirit of nitre, but in similar bottles of amber glass a loss of only 0.06 per cent. occurs under the same conditions during four weeks. Practically the whole of the ethyl nitrite present is lost on exposure in a porcelain basin at the ordinary temperature in 1½ hours. The ideal method of storing sweet spirit of nitre is in small completely filled bottles of amber glass kept in a refrigerator, when no appreciable loss of ethyl nitrite will occur in a month.

T. J. W.

Microchemical Identification of Hydrocyanic Acid by Means of Alloxan Reagent. G. Denigès. (*Ann. Chim. Anal.*, 1921, **3**, 179-182.)—In the presence of minute traces of hydrocyanic acid, characteristic crystals are produced by the interaction of ammonia and alloxan, the hydrocyanic acid acting as a catalyst only. The alloxan reagent is prepared as follows: Two grms. of uric acid are treated with 2 c.c. of nitric acid (sp. gr. 1.38) until nitrous vapours cease to be evolved; 2 c.c. of water are then added, and the liquid heated until clear, and finally made to 100 c.c. For the detection of hydrocyanic acid, a drop of alloxan reagent is placed on a cover-slip, and a minute quantity of ammonia is added immediately; the cover-slip is then inverted over the mouth of a small test-tube

(about 18 × 50 mm.) containing the solution suspected to contain hydrocyanic acid, and allowed to remain for a short time. The test may also be carried out by exposing a drop of dilute ammonia in the same way, and then adding a minute quantity of the alloxan reagent; the substitution of pyridine for ammonia results in a still more sensitive reaction. The test is capable of indicating as little as $\frac{1}{10000}$ of 1 mgrm. of hydrocyanic acid. The mixture of alloxan reagent with pyridine or ammonia must be made immediately before use. The reaction can be used for the detection of hydrocyanic acid derived from cyanogenetic glucosides in beans, etc., by exposing a drop of the reagent for a few hours over the mouth of a test-tube containing a few grains of the material ground in water. R. G. P.

Examination of Some Methods of Ascertaining the Purity of Saccharin.

P. V. McKie. (*J. Soc. Chem. Ind.*, 1921, 40, 150-152r.)—Pure saccharin melts at 227° to 227·5° C. (corr.); study of the melting point curves of mixtures of saccharin *o*- and *p*-sulphamidobenzoic acids and with *o*-toluenesulphonamide shows that the melting-point is a safe guide to the percentage composition of mixtures up to 10 per cent. in the case of the para-acid (m.-pt., 288° to 289° corr.) and the ortho-amide. Mixtures with the ortho-acid cannot be estimated by the melting-point, as this acid decomposes at 180° C. If a 6 to 8 per cent. solution of sodium bicarbonate be added to the powdered saccharin until effervescence ceases, and the mixture filtered after standing, *o*- and *p*-toluenesulphonamides and toluene-2·4-disulphonamide are left behind, and pure saccharin can be precipitated from the filtrate by adding 30 to 50 per cent. excess of 5 per cent. hydrochloric acid at 80° C. When saccharin is estimated by the usual method of hydrolysing with acid and distilling the ammonia, *o* and *p*-toluenesulphonamide and toluene-2·4-disulphonamide are not hydrolysed, and so do not interfere with the accuracy of the method, but *o*-sulphamidobenzoic acid is largely hydrolysed, so that this not very common impurity introduces considerable inaccuracy in the estimation (*cf.* ANALYST, 1919, 44, 99). H. E. C.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Investigations on the Rancidity of Butter and Margarine Fats. W. N. Stokoe. (*J. Soc. Chem. Ind.*, 1921, 40, 75-81r.)—The rancidity of butter and margarine is almost wholly caused by the action of micro-organisms; the first stage appears to be hydrolysis, and this is followed by oxidation and a number of other reactions. The kinds or forms of rancidity are several, and may be classified according to the appearance, odour, and taste of the fat. Oxidation and heat produce a stale greasy taste in the fat; the tallowiness of old butter is another instance of this form of rancidity. Another kind of rancidity is that in which the fat acquires an aromatic odour and a most objectionable pungent taste; this may occur in margarine only a few days old. Investigation showed that mould organisms, principally *Penicillium* species, were present in these rancid margarines, and that the growth of *Penicillium* or of *Aspergillus* on media containing coconut oil or palm-kernel oil produced this form of rancidity; the effect appears to be brought about by an enzyme secreted by the mould acting probably in conjunction with the lipase secreted by the

same organism. The substance produced and giving rise to the odour and taste is a ketone or mixture of ketones (principally methyl-nonyl-ketone); aldehydes are not present. In cases of rancidity where there is a marked discoloration of the butter or margarine, the effect is produced by moulds and also by bacteria and yeasts; the following organisms were isolated from rancid butter and margarine: *B. fluorescens* liq., *B. proteus* species, *B. coli*, *B. putrificus*, *B. punctatum*, *M. luteus*, *S. lutea*, wild yeasts, torulæ, etc. Yeast organisms are found frequently in butter, and their action may be capable of useful development; if a mixture of fats containing coconut oil be inoculated with yeast, a faint, slightly fruity odour is produced. Certain acid-resisting yeasts appear to have the property of associating themselves with the lactic acid organisms, whereby the life of the latter is prolonged.

W. P. S.

Black Spot of Citrus Fruits. H. A. Lee. (*Philippine J. Science*, 1920, 17, 635-641.)—The black spots formed on the rind of oranges, lemons, mandarins, and other citrus fruits, are due to a fungus, *Phoma citricarpa*, which develops in storage and is especially found on oranges grown in China. The fungus may be cultivated on beef agar, glucose agar, or potato, but is frequently overgrown by secondary fungi. The diagnostic features are the black, carbonaceous, restricted growth on culture, and the thin-walled, granular, characteristic spores (9.25 to 12.25×5.5 to 8.1μ). The fungus should produce a growth on sweet oranges in fifteen to twenty days. The disease is common in Australia, but is not yet known in the orchards of America or Japan.

Sudden Physiological Mutations caused by Individual Divergences of Lactic Organisms. C. Gorini. (*Comptes rend.*, 1921, 172, 1382-1384.)—Normally lactico-proteolytic organisms first coagulate milk and then dissolve the coagulum, but occasionally a variety is encountered which peptonises milk without previously coagulating it. This modification, which exhibits diminished acidifying power, transmits its properties, although cases of retromutation—that is, reversion after several generations to the original behaviour—have been observed. It is suggested that the organism contains some cells in which the saccharolytic, and others in which the proteolytic, power predominates, and that the prevalence of the one or the other type in the seeding determines the character of the resulting culture.

T. H. P.

Diagnosis of Individual Blood and Sperm. Dervieux. (*Comptes rend.*, 1921, 172, 1384-1386.)—Five doses of one and the same human sperm containing living spermatozoa were administered subcutaneously to a rabbit at intervals of three days. After the lapse of three weeks blood was withdrawn from the carotid of the rabbit, the serum being collected aseptically and stored in sealed tubes. The behaviour of the serum towards human sperm, male and female human blood, and the blood and sperm of the individual supplying the sperm injected into the rabbit, was investigated, the results obtained showing that it is possible in this way (1) to ascertain if a sperm is of human origin and from which particular individual it was derived, and (2) to determine if any sample of blood is of human origin, to diagnose it as male or female blood, and to ascertain if it is or is not the blood of a

particular individual. If the sperm of different animals be employed, the method may possibly be applied more generally.

T. H. P.

WATER ANALYSIS.

Soap Solution for Use in Estimating Hardness of Water. A. Krieger. (*Chem. Zeit.*, 1921, **45**, 559-560.)—The author recommends the use of a 10 *N* soap solution instead of normal Clark's solution; it is prepared by dissolving 20 grms. of potash soap in 50 c.c. water, and adding 94 per cent. alcohol up to 700 c.c. An appreciable saving of alcohol is effected.

W. J. W.

Estimation of the Hardness of Water. G. Brühns. (*Zeitsch. angew. Chem.*, 1921, **34**, 279.)—Winkler's method (*Zeitsch. angew. Chem.*, 1921, **34**, 115) has been slightly modified: 150 c.c. of the sample are treated with $\frac{N}{10}$ hydrochloric acid, with methyl orange as indicator, until a red coloration persists and free carbon dioxide is expelled. About 0.03 to 0.05 gm. of precipitated calcium carbonate is added, and then 25, 50, or 75 c.c. of Wartha's solution, dependent on the degree of hardness, after which the solution is diluted to 250 c.c. After remaining for half to one hour until two-thirds of the solution have settled clear, a sample is withdrawn and tested for alkalinity.

W. J. W.

Detection of Phenols in Water. R. D. Scott. (*J. Ind. Eng. Chem.*, 1921, **13**, 422.)—The reagent used is that proposed originally by Folin and Denis for the colorimetric estimation of phenols in urine; it is prepared by boiling a mixture of 750 c.c. of water, 100 grms. of sodium tungstate, 18 grms. of molybdic acid, and 50 c.c. of 85 per cent. phosphoric acid for two hours under a reflux condenser. The solution is then cooled and diluted to 1 litre. Five hundred c.c. of the water to be tested are acidified with 10 c.c. of sulphuric acid (1:1) and distilled; 100 c.c. of distillate are collected, and treated with 1 c.c. of the reagent and 5 c.c. of saturated sodium carbonate solution. The distillate from a water containing as little as 0.1 part per million of phenol gives a distinct blue coloration. The distillation removes substances (especially tannin) which interfere, and the test becomes practically specific for phenols.

W. P. S.

Detection of Fluorescein in very Dilute Solutions. M. Lombard. (*Bull. Soc. Chim.*, 1921, **29**, 462-464.)—Fluorescein is commonly used as a powerful colouring matter in the investigation of the contamination of drinking-water. The identification of fluorescein in very dilute solutions may be effected by acidifying 30 c.c. of the water with a few drops of sulphuric acid or of hydrochloric acid (free from chlorine), and shaking with sufficient ether to yield a layer of 3 to 4 mm.; a few drops of ammonia are then added and mixed by gentle shaking, when, in the presence of fluorescein, the ether layer appears green when seen against a dark background; on standing, the fluorescein, which is more soluble in water than in ether, tinges the surface of the aqueous layer. One part of fluorescein in 200,000,000 may be detected. For greater dilutions, 200 c.c. of water are acidified and extracted with

30 c.c. of ether, the ether layer is then separated from most of the water, concentrated by evaporation, and examined after adding ammonia; ¹⁰⁰⁰⁰⁰⁰⁰⁰⁰fluorescein may be detected by this means. R. G. P.

AGRICULTURAL ANALYSIS.

Estimation of Urea in Fertilisers. E. B. Johnson. (*J. Soc. Chem. Ind.*, 1921, **40**, 1267.)—For the estimation of urea, advantage is taken of the fact that it forms a slightly soluble salt with oxalic acid, the solubility of this salt being kept as low as possible by suitable precautions. Two to 5 grms. of the dried sample are extracted with 100 c.c. of anhydrous amyl alcohol, 25 to 50 c.c. of the filtrate treated with an equal volume of ether, and 25 c.c. of a 10 per cent. solution of anhydrous oxalic acid in amyl alcohol added. The precipitate is stirred and allowed to stand in cold water for thirty minutes, after which it is collected on a Gooch crucible, washed with a mixture of equal parts of amyl alcohol and ether, and then with ether alone, dried in a vacuum desiccator, and weighed. The necessary corrections for solubility are indicated by the following table :

Urea found (grms.) :	0.005	0.01	0.02	0.04	0.05	0.08
Percentage of actual amount present :	90.0	92.8	95.2	97.5	98.0	98.8

In cases where the urea is present in the fertiliser as a salt, or in complexes such as $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{CO}(\text{NH}_2)_2$, it will not dissolve completely in amyl alcohol and must therefore first be liberated. W. J. W.

ORGANIC ANALYSIS.

Iodimetric Estimation of Mercaptans. J. W. Kimball, R. L. Kramer, and E. E. Reid. (*J. Amer. Chem. Soc.*, 1921, **43**, 1199-1200.)—About 0.25 gm. of the sample is weighed in a stoppered weighing bottle, and dropped into a bottle containing 35 c.c. of $\frac{N}{10}$ iodine in aqueous potassium iodide solution. The bottle is immediately stoppered and well shaken, and the excess of iodine titrated with $\frac{N}{10}$ sodium thiosulphate solution, starch being used as an indicator. A series of estimations made on various aliphatic mercaptans of approximate purity gave very uniform results with the same sample. T. J. W.

Iodimetric Estimation of Acetone. P. H. Hermans. (*Chem. Weekblad*, 1921, **18**, 348.)—The accuracy of the iodoform method for estimating acetone is dependent on the proportion of potassium hydroxide added to the aqueous acetone solution. Suitable amounts are: For 20 c.c. of acetone solution, 10 to 15 c.c. of $1\frac{1}{2} N$ KOH; and for 100 c.c. of acetone solution, 25 to 30 c.c. of $1\frac{1}{2} N$ KOH. In the first case the conversion into iodoform is complete in one to two minutes, and in the second case in three to five minutes. In an experiment with 0.2353 gm. of acetone in 100 c.c. water, 10 c.c. diluted to 100 c.c., and treated with 30 c.c. of potassium hydroxide solution and 25 c.c. of $\frac{N}{10}$ iodine solution, gave 99.9 per cent. acetone. In a second experiment, 10 c.c. were diluted to 20 c.c. and to 80 c.c. respectively, and 10 c.c. were distilled and diluted to 100 c.c.; the amounts of hydroxide added in the

three cases were 12, 25, and 30 c.c., and the iodine was added as before. After acidifying and titrating with thiosulphate, 99.5 per cent. of acetone was obtained in each case.

W. J. W.

Direct Estimation of Dicyanodiamide. E. B. Johnson. (*J. Soc. Chem. Ind.*, 1921, 40, 125-126t.)—A volumetric method for the estimation of dicyanodiamide is based on the precipitation of a compound of silver picrate with 2 mols. dicyanodiamide (cf. Harger, *J. Ind. Eng. Chem.*, 1920, 12, 1107). Five grms. of the sample, or a correspondingly larger amount if the nitrogen be less than 5 to 15 per cent., are treated with 450 c.c. of cold water, glacial acetic acid being added to dissolve lime if present. The mixture is shaken for three hours, diluted to 500 c.c., and filtered. To 100 c.c. of the filtrate are added 5 c.c. of 20 per cent. nitric acid and 20 c.c. sodium picrate solution at 40° C.; the solution is then cooled to 5° C. and titrated with $\frac{N}{25.4}$ silver nitrate solution, of which about 2 c.c. are added in excess of that required by the amount of dicyanodiamide believed to be present. After vigorous shaking, the mixture is allowed to stand for fifteen minutes at 5° C., diluted to 200 c.c. with cold water, and filtered. Excess of silver solution is then titrated with $\frac{N}{22.4}$ sodium thiocyanate solution, after addition of 5 c.c. of 20 per cent. nitric acid and 2 c.c. of 5 per cent. ferric sulphate solution as indicator. With a 5 gm. sample, each c.c. of silver solution corresponds to 1 per cent. of nitrogen as dicyanodiamide. The results are affected by the excess of silver solution or the amount of nitric acid used, whilst temperature has also an influence on the precipitation. Suitable corrections must therefore be worked out, but none is necessary in the case of calcium cyanamide if 5 c.c. of 20 per cent. nitric acid per gm. of calcium acetate be used, together with an excess of 2 c.c. of silver nitrate. The method is not affected by the presence of urea and dicyanodiamidine; if chlorides and soluble sulphides be present, a blank titration of the silver solution without addition of picric acid must be made. The results agree closely with those obtained by the modified Caro method.

W. J. W.

Estimation of Fatty Acids in Turkey-Red Oil. (*Chem. Zeit.*, 1921, 45, 560-561.)—For the volumetric estimation of the fatty acids in Turkey-red oil, 10 to 20 grms. of the sample are heated with 25 c.c. of water until dissolved. The solution is transferred to a Büchner flask, 30 c.c. of concentrated hydrochloric acid are added, and the mixture is heated for twenty minutes over the naked flame. Sufficient concentrated solution of sodium chloride at 100° C. is then added to cause the fatty acid layer to enter the tube of the apparatus, and, after fifteen minutes on the water-bath, the volume of fatty acids is read off, the percentage by weight being calculated by means of a specific gravity determination. For the gravimetric estimation, 10 grms. of the sample are dissolved in 50 c.c. water in a porcelain crucible on a water-bath, 15 c.c. concentrated hydrochloric acid are added, and the mixture is left for thirty minutes, during which period the layer of fatty acids is agitated with the acid liquid. When the fatty acids have separated clearly, 10 grms. of wax are added, and the mixture is allowed to remain on the water-bath for thirty minutes with agitation, after which it is left to settle for an equal period, and cooled. The wax is then re-

moved, rinsed, melted in distilled water, solidified and dried, heated in a crucible for thirty minutes at 105° to 110° C., and weighed. The weight of fatty acids is then calculated.

W. J. W.

Estimation of Resin Acids in Fatty Mixtures. D. McNicoll. (*J. Soc. Chem. Ind.*, 1921, 40, 124r.)—The author describes a method for the estimation of resin acids in which the inaccuracies of Twitchell's method are avoided, whilst at the same time simplicity is gained by substitution of methyl for ethyl alcohol and of naphthalene- β -sulphonic acid for hydrogen chloride. In a volumetric estimation, 2 grms. of the mixture are dissolved in 20 c.c. of a 4 per cent. solution of naphthalene- β -sulphonic acid in methyl alcohol and heated for thirty minutes under a reflux condenser on an electric plate or oil-bath, a blank experiment with the sulphonic acid solution being carried out simultaneously. The contents of the flasks are then cooled and titrated with $\frac{N}{2}$ solution of potassium hydroxide in methyl alcohol. The resin acids are calculated on the combining weight of 346. For a gravimetric estimation, the neutralised solution of known volume obtained above is transferred to a separating funnel and extracted with an equal volume of ether and petroleum spirit; the soap solution is similarly extracted, the combined extracts are washed with 50 per cent. aqueous alcohol, and the washings added to the resin soap solution, which is acidified and extracted with ether. Pure fatty acids are completely esterified by this process; when it is applied to resin, partial esterification, to the extent of 1 to 3 per cent. of the resin acids, takes place. With three mixtures containing 11.51, 29.84, and 48.69 per cent. of resin, respectively, the results obtained by the volumetric method were 11.84, 30.28, and 48.45 per cent.; and by the gravimetric estimation, 11.73, 30.23, and 48.88 per cent. In a series of tests the errors varied between -0.22 and -0.56 per cent. in the volumetric method, and between -0.38 and +0.35 per cent. in the gravimetric method. Lewkowitsch has shown that by Twitchell's method the errors vary between -9.73 and +5.26 per cent.

W. J. W.

Apparatus for Estimating the Yield of Tar from Coal. H. Schrader. (*Brennstoff. Chem.*, 1921, 2, 182-183.)—A modification of the apparatus for the estimation of tar in coal (*Brennstoff. Chem.*, 1921, 1, 87; *J. Soc. Chem. Ind.*, 1920, 566A) is described by which steam used in the process is superheated in the apparatus itself. With this object, the supporting arm walls and base of the apparatus are drilled, so that steam introduced through the arm passes vertically downwards, traverses the base in a V-shaped course, and then passes upwards and through the cover into the interior of the retort. Suitable apertures in the retort walls to facilitate cleaning of the steam channels, and means for removing condensed water, are provided. In order to prevent condensation in the retort, steam is not introduced till the temperature has reached 130° C., and the rate of the current is controlled so as to avoid mechanical loss of coal. A small condenser is inserted between the delivery tube and the receiver; the heavy oils which collect in the condenser are separated by treatment with ether, whilst lighter oils are similarly recovered from the final receiver.

W. J. W.

The Setting and Melting Points of Gelatins. S. E. Sheppard and S. Sweet. (*J. Ind. Eng. Chem.*, 1921, **13**, 423-426.)—In the apparatus described an intermittent stream of air bubbles under constant pressure is passed through the test solution, the latter being cooled with ice-water. A thermometer is immersed in the solution with its bulb adjacent to the air passage, and the temperature at which the bubbles cease to pass is taken as the setting-point. Inversely, after sufficient under-cooling, the set jelly is heated gradually, and the melting-point taken as the temperature at which bubbles again pass through the solution. The compressed air employed passes a manometer and a manostat bottle, and enters a U-tube containing mercury, which serves as a valve to produce intermittence in the air supply; a solenoid, the current through which is made and broken by a timing device every fifteen seconds, effects this interruption by operating an iron plunger. From the U-tube the air passes to a compensating U-tube, and from this to the setting-point tube. The gelatin solutions (1, 3, 5, 10, 15, and 20 per cent. air-dry substance) used are prepared under standard conditions. Results recorded show that the relation between melting-point and concentration is only approximate, and that a similar restriction is true as regards the relationship to jelly strength. W. P. S.

INORGANIC ANALYSIS.

Poirrier Blue as an Indicator. W. Mestrezat. (*J. Pharm. Chim.*, 1921, **23**, 489-494.)—Experiments with a number of samples of this indicator show that the colour changes occurring in the titration of alkali or of mixtures of alkali and alkaline carbonate are progressive, and so much affected by dilution that this indicator compares unfavourably with methyl orange or phenolphthalein. R. G. P.

Modified Iodine Solution for the Reich Test. H. M. Lowe. (*J. Soc. Chem. Ind.*, 1921, **40**, 123-124r.)—In the standard iodine solution used for estimating sulphur dioxide in gases the potassium iodide may be replaced by a proportion of sodium hydroxide insufficient to react with the whole of the iodine, but sufficient to form enough sodium iodide to dissolve the remaining iodine. From a solution consisting of iodine, 127 grms.; sodium hydroxide, 30 grms.; and water, up to 10 litres, the theoretical amount of iodine is always found to be liberated on acidification. With a $\frac{N}{10}$ solution no further addition of acid is necessary; but when dilutions of $\frac{N}{100}$ to $\frac{N}{2000}$ are employed, as in testing exit gases, a small amount of sulphuric acid or hydrochloric acid must be added to cause the iodide and iodate in the solution to react instantaneously. W. J. W.

Detection and Estimation of Traces of Hydrogen Peroxide. F. W. Horst. (*Chem. Zeit.*, 1921, **45**, 572.)—Traces of hydrogen peroxide may be estimated by reduction with ferrous sulphate solution, and subsequent colorimetric estimation of the ferric sulphate by means of ammonium thiocyanate. In order to obtain the ferrous sulphate solution free from ferric salts, it is placed in a closed Erlenmeyer flask and a current of hydrogen sulphide is passed through it, first in the cold and then during boiling; the solution is then cooled in a current of carbon dioxide.

Twenty c.c. of the sample are placed in a tube, and a few c.c. of petroleum spirit added to form a protective layer against oxidation during the reaction. After addition of 2 c.c. of the ferrous sulphate solution, the mixture is agitated by means of a stream of carbon dioxide freed from oxygen by passing it through ferrous sulphate solution, and 5 c.c. of concentrated ammonium thiocyanate are introduced. After further agitation, the colour is compared with that of a standard solution.

W. J. W.

Estimation of Silver by Precipitation with Hypophosphorous Acid. L. Moser and T. Kittl. (*Zeitsch. anal. Chem.*, 1921, 60, 145-161.)—Soluble silver salts are readily reduced to colloidal silver by hypophosphorous acid, and this reaction may be employed for their quantitative estimation. At 20° C. the solubility of the silver gel in water was found to be 4×10^{-5} g.-ions per litre; in $m/40$ and $m/400$ solutions of hypophosphorous acid the solubility is highest at 50° C., the figures being 4.2×10^{-5} and 1.1×10^{-4} g.-ions per litre respectively. Analysis of the reaction product after reduction gave, in four experiments, 7.1 to 8.5 per cent. H_3PO_4 , 39.6 to 43.8 per cent. H_3PO_3 , and 48.6 to 51.9 per cent. H_3PO_2 . The silver colloid contained traces of adsorbed phosphoric acid. The method gives good results when applied to the separation of silver from lead, zinc, or cadmium.

W. J. W.

Separation of Silicon, Tin, Titanium, and Zirconium by Means of Sodium Carbonate. P. Wenger and J. Morel. (*Ann. Chim. Anal.*, 1921, 3, 139-142.)—A mixture of the four oxides is ignited, weighed, and then fused with six times its weight of sodium carbonate, the mixture being first heated just to fusion for ninety minutes, and then for the same period over a blowpipe flame. When cold, the fused mass is boiled with water, the solution filtered, and the insoluble portion washed; the filtrate, which contains the silica and a portion of the tin, is evaporated with nitric acid, the residue of silica and metastannic acid collected, ignited, and weighed, and the silica then separated by means of hydrofluoric acid. The insoluble portion from the sodium carbonate fusion contains the remainder of the tin, and the zirconium and titanium oxides; it is ignited, weighed, fused with eighteen times its weight of potassium bisulphate, and the fused mass boiled with water acidified with nitric acid. Metastannic acid remains insoluble, and is collected on a filter, ignited, and weighed; the filtrate is neutralised, the zirconia precipitated by the addition of hydrogen peroxide, collected, and weighed. Titanium is then precipitated from the solution by ammonia.

W. P. S.

Reductions with Zinc and Cadmium in Volumetric Work. W. D. Treadwell. (*Helv. Chim. Acta*, 1921, 4, 551-565.)—The use of Jones's reductor for the reduction of ferric salts was investigated; it was found that reduction was complete with a 2.5 cm. column of granulated zinc if the ferric solution was admitted drop by drop. Using a 9.5 cm. column, complete reduction was attained, even on rapid percolation. A moderate degree of acidity favours the rate of reduction, whilst precipitation of metallic iron need not be feared in solutions containing 2 per cent. of sulphuric acid. The risk of precipitating metallic iron is quite obviated by the use of cadmium as a

reducing agent. A 5 cm. column of crystalline electrolytic cadmium free from sponge gave rapid and complete reduction with various degrees of acidity, and a small cadmium consumption. A preliminary investigation was made regarding the efficiency of the cadmium reductor in the volumetric estimation of titanium, molybdenum, vanadium, and uranium; very promising results have so far been obtained.

W. R. S.

Estimation of Total Carbon and New Method for Graphitic Carbon in Ferro-Alloys. P. Wenger and A. Trampler. (*Helv. Chim. Acta.*, 1921, 4, 547-551.)—The total carbon was determined in various ferro-alloys by combustion in an electric furnace at 1,150° C., and by Corleis' method (chromic-sulphuric acid mixture). It was found that the results obtained by combustion were higher and more concordant; combustion is also quicker, and applicable to any ferro-alloy; hence it is the best practical method. In the combustion of ferro-alloys of chromium, molybdenum, silicon, and manganese, the use of an oxidiser is necessary; bismuth sesquioxide is recommended. Sulphur dioxide is retained in a heated porcelain tube containing lead chromate. Graphitic carbon may be estimated in ferro-manganese, -vanadium, -chromium, and -silicon by digesting 1 gram. of the powdered alloy with 100 c.c. of phosphoric acid of sp. gr. 1.7; the acid is first heated to 150° C. in a platinum dish; the powder is then gradually added. The heat is raised to 230° to 250°, the acid is decanted, and the attack completed with 25 c.c. of fresh acid. The undiluted liquid is filtered on a Gooch crucible, and the residue is washed with 300 c.c. of water, dried, and treated by combustion in an electric furnace. In the case of ferro-silicon, hydrofluoric acid must be added to the phosphoric acid, but even this mixture fails if the silicon exceed 60 to 65 per cent.

W. R. S.

Volumetric Estimation of Aluminium in its Salts. A. Tingle. (*J. Ind. Eng. Chem.*, 1921, 13, 420-422.)—Investigation of various procedures which have been suggested for the titration of aluminium salts with alkali solution, using phenolphthalein as indicator, showed that two only are reliable. The first consists in titrating the boiling solution containing the aluminium salt with $\frac{N}{2}$ sodium hydroxide solution until the pink coloration obtained with the phenolphthalein added persists after boiling for one minute. In the second method, which is particularly applicable to aluminium sulphate, 100 c.c. of a 1 per cent. solution of the salt are boiled, treated with 5 c.c. of saturated barium chloride solution, and then titrated while hot, as described. The advantage attained by the addition of the barium chloride is that basic aluminium chloride is less stable and more soluble than the basic sulphate, and, further, the precipitated barium sulphate completely masks the colour of iron salts if these be present. It is important that the concentration of the alkali solution used should not be greater than $\frac{N}{2}$. If the titration be carried out on the cold solution, basic salts form and interfere, and the end-point is obscured because the precipitate does not settle readily. There is no advantage in the use of barium hydroxide solution in place of sodium hydroxide solution.

W. P. S.

Production and Testing of Zirconia. W. R. Schoeller. (*J. Soc. Chem. Ind.*, 1921, 40, 127-128r.)—The method of extracting zirconia from Brazilian ores, based on precipitation of zirconium as basic sulphate (Rossiter and Sanders, *J. Soc. Chem. Ind.*, 1921, 70r), does not eliminate titanium, which is present to the extent of 0.6 to 1.2 per cent. It may be completely removed by recovering the zirconium as oxychloride, but this operation is more expensive. A combined oxychloride and basic sulphate method is considered by the author to repay further investigation. The ore is fused with sodium carbonate and extracted with hot water, the recovered alkali being used for treating further ore. The insoluble residue is treated with the zirconium oxychloride wash-liquors, the solution evaporated to dryness, and the mass extracted with hot water. After filtration, the filtrate is mixed with hydrochloric acid and crystallised; the crystals are filtered off, washed with hydrochloric acid, heated to expel water, and finally boiled with dilute ammonia and ignited. The acid used for washing the oxychloride, mixed with some of the oxychloride mother-liquor, is used again to dissolve the fused residue, whilst the remainder of the mother-liquor is neutralised and treated with sulphurous and sulphuric acids. The zirconia obtained is free from iron and alumina; for removing titania, fractional solution or precipitation may prove suitable. Commercial zirconia may contain, in addition to iron, aluminium, and titanium, appreciable amounts of silica, arsenic, and sulphite; in some samples, lime, sodium carbonate, fluorine, chlorine, carbon dioxide, and boron trioxide, have been found. For the estimation of arsenic, 2 grms. are fused with 15 to 20 grms. of sodium carbonate, extracted with hot water, acidified, and treated with hydrogen sulphide, preferably after addition of potassium iodide to reduce arsenious acid. The sulphide is then evaporated with sulphuric acid till fumes are evolved, and the liquid is made alkaline with bicarbonate and titrated with iodine solution.

W. J. W.

Gasometric Estimation of Hypochlorites. A. K. Macbeth. (*Chem. News*, 1921, 122, 268.)—Hypochlorites may be accurately estimated by treating them in a Van Slyke nitrometer (*J. Biol. Chem.*, 1912, 12, 278), with an alkaline solution of hydrazine (prepared by dissolving hydrazine sulphate and potassium hydroxide in water), and measuring the nitrogen evolved. Free chlorine must be absent. In one set of experiments this method gave 16.13 grms. potassium hypochlorite per litre, as compared with 16.063 and 16.11 grms. by titration with arsenious acid and sodium thiosulphate respectively; further comparative results by the three methods were 23.55, 23.54, and 23.55. The method may be used for estimating available chlorine in bleaching powder. It has not given satisfactory results when applied to the estimation of chlorates.

W. J. W.

Examination of Detonators and Percussion Caps. A. Langhans. *Zeitsch. ges. Schiess.- u. Sprengstoffw.*, 1921, 16, 49-52, 57-59.)—Detonator and percussion-cap compositions containing as ingredients mercury fulminate, potassium chlorate, antimony sulphide, powdered glass, gunpowder, and shellac, may be identified by a microscopic examination, as well as, to some extent, by their behaviour with concentrated acids. For the quantitative examination a method is described in which both

the composition and its container are brought into solution and then submitted to analysis; by this means any risk involved in removing the composition from the shell for testing is eliminated. About four percussion caps are dissolved in a mixture of 50 c.c. of nitric acid and 25 c.c. of water. The solution is evaporated to dryness, and the residue, after boiling three times with dilute nitric acid, is filtered, by which means powdered glass, together with some charcoal and sulphur, is separated. After dilution of the filtrate to 500 c.c., 100 c.c. are treated with hydrogen sulphide, and the sulphides of mercury and antimony in the precipitate are dissolved by treatment with sodium sulphide solution and potassium hydroxide; the residual copper sulphide is removed by filtration, dissolved in acid, and electrolysed. The filtrate is acidified, and the antimony sulphide separated by treatment with ammonium sulphide and precipitation with sulphuric acid; the mercury sulphide is dissolved in nitric acid and electrolysed. Chlorate and nitrate of potassium are estimated in the original filtrate from the hydrogen sulphide precipitation. With detonators containing a mixture of potassium chlorate and fulminate of mercury, a similar method may be applied; estimation of the chlorate by the use of nitron is also suitable.

W. J. W.

PHYSICAL METHODS, APPARATUS, ETC.

Apparatus for Microscopic Examination of Opaque Objects. M. Francois and C. Lormand. (*Bull. Soc. Chim.*, 1921, 29, 366-374.)—Opaque objects are illuminated by rays of light focussed on the object from a small concave mirror of polished silver or white metal (about 1 mm. in thickness) fixed to the end of the objective, and pierced by a central hole (about 1 mm. in diameter) through which the object is viewed; each objective requires a mirror of such curvature that the light is accurately focussed on the object when the latter is in focus on the microscope stage. An electric lamp (2 to 3 volts) with a bulb of lenticular shape (about 2 cm. in diameter), having a metallic filament of lozenge or S-form, and mounted in a tube with a lens to render the rays parallel (an eyepiece without the lower lens), is fixed below the stage, so as to throw parallel rays of light on to the mirror. The light is prevented from passing directly from this source to the objective by mounting the object on a dark-ground of such dimension as to obscure the field visible with any particular objective. The dark-grounds are conveniently made by cutting thin discs from rods (0.75 to 3.0 mm. in diameter) composed of a mixture of kaolin and ground quartz; the discs are then fired at red heat, and (after colouring any shade required) are mounted on a slide with Canada balsam. Examination of objects by this apparatus is best conducted in a dark room. A small electric lamp, as described above, as a source of illumination is suitable for transparent objects, and it is suggested that it may conveniently replace the ordinary reflecting mirror, as the illumination is constant and easily regulated by interposing screens of paper between the source of illumination and the slide.

R. G. P.

Use of Polarised Light for the Examination of Old Pictures. P. Lambert. (*Comptes rend.*, 1921, 172, 1476-1477.)—Even under the best conditions, the image diffused by the colours of old pictures is always attenuated and whitened by the

light reflected from the surface. If, however, the picture be illuminated by means of polarised light, and examined through a Nicol prism, the reflected light will be extinguished if the prism be in the position of extinction; whereas that which traverses the varnish is depolarised at the surface of the materials composing the colours, and is thus able to reach the eye. The method gives results of value in the judging of old pictures, and in indicating a possible improvement by modification of the varnish.

T. H. P.

New Method of Measuring Electrolytic Conductance. C. Marie and W. A. Noyes, jun. (*J. Amer. Chem. Soc.*, 1921, **43**, 1095-1098.)—The hydrogen electrodes used consisted of vertical glass tubes connected at the lower end with a small glass box, one side of which was made of fine platinum gauze and connected with the Wheatstone bridge by a platinum wire. A current of purified hydrogen, which was bubbled through a solution having the same composition as that under examination, was passed down the tube, and escaped through a small aperture in the bottom of the box. The electrodes were immersed in the solution, adjusted to equal potential by carefully regulating the gas pressure in each, and a direct current passed for only a sufficient length of time to indicate the direction of deflection of the galvanometer. A series of determinations, using different inorganic and organic acids and sodium chloride solutions of various strengths, gave results in exact agreement with those obtained by the authors using Kohlrausch's alternate current method, except with sulphuric and hydrochloric acid solutions of greater concentration than $\frac{N}{2}$.

T. J. W.

Rapid Method for Measuring the Auto-discharge of an Electroscope in the Estimation of Radium Emanation. P. Loisel. (*Comptes rend.*, 1921, **172**, 1484-1486.)—In the ordinary method for measuring radium emanation, the discharge current, I_a , of the electroscope is first observed, the emanation being then introduced, and the true intensity of the maximum current, I_m , measured three hours later, calculated by subtracting the former from the latter current. Since the auto-discharge current of the electroscope is subject to considerable variation, particularly in the neighbourhood of radioactive sources, this method may lead to erroneous results. Sensible error may be avoided as follows: The intensity, I_1 , of the ionisation current produced in the condenser is measured three hours after the introduction of the emanation: $I_m = I_1 - I_a$. The condenser is then rapidly exhausted and filled with inactive air; the emanation being removed, only the induced activity remains, and the current produced is $0.57 I_m$. If the ionisation current fifteen minutes after the evacuation be I_2 , $0.285 I_m = I_2 - I_a$, so that $I_a = \frac{I_2 - 0.285 I_1}{1 - 0.285}$.

T. H. P.



NEW BRITISH CHEMICAL STANDARD STEELS.

(ANALYTICALLY STANDARDISED TURNINGS.)

Two new plain carbon steel standards are now ready for issue—viz., "M," needed for some time mainly for colour carbon tests round about 0.23 per cent.; and "01," which fills the vacancy for a colour carbon standard of about 0.33 per cent., in addition to being available for the other elements shown below.

The analyses have been undertaken, as usual, by a number of experienced chemists representing the following interests: British Government Department; U.S. Bureau of Standards; Referee Analysts, independent; Railway Analysts, representing users issuing specifications; and Works Analysts, representing makers and users.

The standard figures ("M" and "01") are as follows:

Carbon, 0.228 and 0.335; silicon, 0.057 and 0.162; sulphur, 0.04* and 0.032; phosphorus, 0.04* and 0.031; manganese, 0.58* and 0.617; arsenic, 0.024; chromium, 0.017; copper, 0.037; nickel, 0.162.

The standards may be obtained either direct from Organising Headquarters, 3, Wilson Street, Middlesbrough, or through any of the laboratory furnishers, at a price just sufficient to cover the cost. A certificate giving the names of the analysts co-operating, the types of methods used, and a detailed list of their figures will be supplied with each bottle.



REVIEWS.

PHYSICAL AND CHEMICAL CONSTANTS. By G. W. C. KAYE, O.B.E., D.Sc., and T. H. LABY, M.A. Pp. 161. London: Longmans, Green and Co. 1921. Price 14s. net.

The fact that this is the fourth edition since the appearance of the first edition in 1911 is sufficient proof alone of the value of this book. The new edition follows closely the lines of earlier editions, but various alterations and additions have been made in the sections dealing with such questions as the figure of the earth and the absolute determination of the acceleration of gravity, whilst the chemical data have been recalculated on the basis of the 1920-21 atomic weights. The tables of physical constants of chemical compounds have been altered and enlarged.

This book contains a very large amount of data to which the research chemist, and particularly the physical chemist, needs to refer, and the covering of a very wide field in a conveniently condensed form renders it most useful for reference purposes, whilst the very copious references to standard works and original papers enhances its value.

R. G. PELLY.

TABLES OF REFRACTIVE INDICES. Vol. II. Oils, Fats, and Waxes. Compiled by R. KANTHACK, edited by J. N. GOLDSMITH, Ph.D., M.Sc., F.I.C. Pp. 295. London: Adam Hilger, Ltd. 1920. Price 25s. net.

Vol. I. of this work, which dealt with essential oils, has been reviewed in this journal (1919, 44, 306), and Vol. II, which is now before us, is compiled on similar lines.

* Approximate.

The compilation includes the results of some 1,750 measurements on over 500 oils, fats, and waxes, and gives approximately 2,500 references to the original literature. The compiler in his wisdom has clearly pointed out in the Preface that it is not within his province to accept or reject data, and thereby he shows that he is well aware that quite a number of published figures of refractive indices are not worth the printer's ink. These worthless figures, like the tares and wheat, have been allowed to grow side by side with reliable ones in scientific literature, and the compiler has gathered them together in this work, leaving the reader to cast out the tares, the weeding out of which is now rendered less difficult by comparison with the many reliable figures so conveniently tabulated.

The collection of data distributed over such a wide expanse of literature cannot fail to impress one as to the patience which such a task involved, and such a collection in a form so convenient for reference will appeal to the practical analyst.

E. R. BOLTON.

THE CHEMISTRY OF SYNTHETIC DRUGS. By PERCY MAY. Third Edition. Pp. xii + 248. London: Longmans, Green and Co. 1921. Price 12s. 6d. net.

In the first forty-three pages of this book Dr. May gives an admirable outline of the relation between chemical constitution and physiological action; in easy style the present knowledge is summarised, and if the conclusions are somewhat incomplete, it is due to the fact that only the fringe of the subject has yielded to investigation.

The remainder of the work is devoted to the chemistry of synthetic drugs, and the author is singularly happy in the manner in which he has classified them; it is practically impossible to find any classification in which no overlapping occurs, but that adopted affords the minimum of such overlapping. The plan adopted is to give the chemical constitution of each substance, with a brief but sufficient indication of its action, with useful comparisons of the difference in action of allied substances.

Little or no attempts are made to give the mode of preparation of the drugs, and the analytical characters are omitted, the author sticking entirely to the chemistry of the subject with a complete absence of padding. A careful search has failed to reveal the absence of any synthetic drug of any importance, and, indeed, the only noticeable omissions are those of alternative names such as *glusidum*, the B.P. name for *saccharin*, and *uradal*, given in the B.P.C. for *adalin*.

There is no doubt that public analysts and those engaged in the examination of drugs who wish to keep their knowledge of the chemistry of the substances they examine up to date will find this book invaluable.

H. DROOP RICHMOND.

THE CHEMISTRY AND ANALYSIS OF DRUGS AND MEDICINES. By HENRY C. FULLER. Pp. 1072. New York: John Wiley and Sons. 1920. Price 55s. net.

Published works on the analysis of drugs and medicines are comparatively few. The analyst engaged in purely pharmaceutical analysis has to draw his textbook information from a wide range of works, none of which deal with drugs as the chief subject. Apart from sections of Allen's "Commercial Organic Analysis" and certain

chemico-pharmaceutical works, such as Squire's "Companion," we have in this country to rely on works in which drugs take second place to foods. It was, therefore, with a hope that this gap would in some measure be filled that the reviewer turned to the present work.

The book under review is divided into five parts as follows:

- I. General Methods and Crude Drug Assays.
- II. Alkaloidal Drugs, Alkaloids, and Medicinally Allied Substances.
- III. Glucosides, Glucosidal Drugs, and Natural Drugs containing Principles other than Alkaloids.
- IV. Organic Substances other than Alkaloids and Glucosides.
- V. Inorganic Section.

It may be said at once that this work is mainly devoted to the analysis of naturally occurring and synthetic organic drugs. The inorganic section of the work occupies only a small space by comparison. Whilst acknowledging that the classification of drugs for a book of this kind is a difficult matter, it must be said that the arrangement of the book is certainly open to criticism; in fact, the more one seeks to discover the basis of the arrangement, the more illogical does the latter appear. In dealing with this point, it is well to inquire from what standpoint the author has written. The predominant idea of the book seems to be the analysis of unknown remedies and the determination of their constituents, whilst the determination of the purity of drugs is a secondary consideration. For this reason the introductory chapter on General Methods deals with the following at first sight somewhat curiously chosen determinations—viz.: Specific Gravity, Ash, Alcohol, Cane Sugar, Glucose Syrup, and Arsenic. There follows a chapter on Crude Drug Assays, chemical and physiological; the greater portion consists of a reprint of the U.S. Pharmacopœia methods, and in many cases both the methods of the eighth and ninth revisions are given, but without any expression of opinion as to which is the better of the two. It is not easy to see why the drugs here included are thus separated from their fellows. The determination of morphine in opium and tincture of opium is to be found in this chapter, but powdered opium, paregoric, and laudanum are found in Chapter VII. Natural drugs are divided into the following: (1) Alkaloidal Drugs, (2) Glucosidal Drugs, (3) Purgative Drugs, (4) Miscellaneous Acting Drugs, and (5) Botanical Drugs. The title of the fourth division might have been more happily expressed, but the meaning of the fifth is distinctly puzzling. It might be meant to include those drugs that do not "act," but on inspection it is found to include *Cannabis Indica*, *Leptandra*, and many others not by any means innocuous. The inclusion of anæsthetics such as chloretone in the chapter on alkaloids derived from pyrrolidin, of columbin under alkaloids derived from isoquinoline, of kino among gums and resins, seems hardly justifiable.

The best feature of the book, and the most valuable to analysts generally, is the inclusion of descriptions of a large number of synthetic and proprietary remedies; many of them, it is true, are unknown or little used in this country, but much useful information is scattered throughout the book. The notes on the uses of the various drugs and the combinations in which they are most likely to be found should also be of great value to the analyst.

The sections dealing with alkaloids and glucosides are on the whole full and well written.

Essential oils and fixed oils are lightly treated, occupying together only twenty-four pages, so that naturally there are many omissions. Cod-liver oil is fully treated, but the limits of specific gravity seem very narrow—viz., 0.9196 to 0.922 at 25° C. In discussing the therapeutic value, no reference is made to what is now known to be the chief factor in the peculiar virtue of cod-liver oil, its high content of fat-soluble vitamin.

The analytical methods are chiefly drawn from American sources; many of them are probably unfamiliar, by name at any rate, to English readers. From an examination of the familiar methods included, it does not appear that any great discrimination has been used in their selection—e.g., the discredited lead number test for asafetida is given in spite of the work of Harrison and Self (*Pharm. J.*, 1913, 218). On p. 653 it is stated that "there is no method for determining the relative quantities of citric and tartaric acids in a mixture of the two"; on p. 650 we find, "when tartaric acid occurs simultaneously with citric acid, the potassium method will give good results"; and on p. 655 is given the useful mercury method of Gowing Scopes (*ANALYST*, 1913, 38, 12) for the same purpose.

On p. 491, evidently by a misprint, -70 is given as the minimum rotation of copaiba oil.

On p. 750 the solubility of calcium glycerophosphate is erroneously given as 1:400.

To sum up, it may be said that the book will provide much useful information to the analyst who has to deal with unknown mixtures of drugs or medicines. Its value to the analyst in a pharmaceutical works, or to one who is primarily engaged in assaying or testing the purity of drugs, is considerably less; for though he may turn to it occasionally for information, he is hardly likely to use it as a standard textbook of drug analysis.

NORMAN EVERS.

ANIMAL AND VEGETABLE FIXED OILS, FATS, BUTTERS, AND WAXES. By C. R. ALDER WRIGHT. Third Edition, revised and greatly enlarged by C. AINSWORTH MITCHELL. Pp. 939. London: Charles Griffin and Co., Ltd. 1921. Price 56s. net.

The second edition of this work, published in 1903, has been out of print for the last two years, but earlier revision has been prevented by the war. As the reviser states, the literature of the subject has multiplied very greatly during the past few years, and it has become necessary to make a selection from the mass of material available. Even so, the volume has increased from 804 to 939 pages, partly due to the fact that the section on margarine has been rewritten, and a new chapter on hydrogenated oils added; while the International Standard methods for glycerine analysis are incorporated in the last chapter, and an official report from the Ministry of Food on Standard Methods of Analysis of Seeds, Nuts and Kernels, Fats and Oils, and Fatty Residues, and a summary of recent work on vitamins in relation to fats, are included as appendices.

In the preparation of the second edition the aim was to fit it particularly for use as an analytical textbook, and this characteristic of the work is still maintained, both as regards raw materials and the manufactured products, the recorded values for the individual oils being brought up to date, and the more recently introduced analytical processes described. It is a pity that, although such useful methods as those of Polenske and Kirschner are given fairly fully, just sufficient data are omitted to render it impossible for the novice to carry out the determinations without seeking information elsewhere, and it is surely time that the phrase "a few fragments of pumice" should disappear from the Polenske process, and give place to the correct "0.1 gm. of powdered pumice." Probably few analysts would consider ten minutes' contact long enough for determining the iodine value of linseed oil by the Wijs method, two hours, as prescribed by the British Pharmacopœia, being much more satisfactory. Under Chinese wood oil, Hoepner and Burmeister are quoted as getting untrustworthy results by the methods of Wijs and Hanus, but by adopting Chapman's method (ANALYST, 1912, p. 545), dissolving 0.1 gm. of oil in 20 c.c. carbon tetrachloride, adding 30 c.c. Wijs' solution, and allowing to stand for three hours before titration, perfectly satisfactory results may be obtained. No reference is made to the Ave-Lallemant method of examining butter-fat (ANALYST, 1907, p. 382), and it is surprising in a book of this character to find the only method given for estimation of boric acid is that of distillation with methyl alcohol.

The description of the individual oils and fats covers all the more important varieties, but it is disappointing to find only carnauba wax under the "vegetable non-glyceridic waxes," no mention being made of candelilla, esparto or reed, and sugar wax, although these are all of technical importance. The expression "non-glyceridic wax" itself appears open to objection, as all true waxes are necessarily non-glyceridic, and it is rather regrettable that this distinction between fats and waxes is not made more clear both in the introductory chapter and in the classification on p. 370.

The statement on p. 692 that bees fed on sugar are incapable of developing wax to any notable extent is incorrect, several experimenters having shown that bees fed on sugar produce more wax than those fed on honey.

The chapter on hydrogenation of oils outlines the more important patents for plant, preparation of catalyst, and hydrogenation process. The properties of hardened oils are described, and the dimethyl-glyoxime test is given as the best for the detection of nickel, but no mention is made of the extremely sensitive α -benzildioxime method for the detection and estimation of this metal. The sections on technology remain much as before, descriptions of the Twitchell and castor ferment methods for fat-splitting being now included, and also modern plant for the concentration and distillation of glycerin. The rewritten section on margarine gives a useful and up-to-date review of the modern developments in this industry.

The book is well printed and illustrated, though one or two of the blocks in the soap section are a little out of date. Owing to an obvious error in setting up on p. 502, the description of rape oil appears under radish-seed oil, but there are very few misprints, only two or three trivial ones being detected. The reviser is to be congratulated on the vast amount of information he has brought together within the confines of a single book.

W. H. SIMMONS.

REPORTS OF THE PROGRESS OF APPLIED CHEMISTRY. Vol. V. 1920. Issued by the Society of Chemical Industry. Price 8s. 3d. to members, 15s. to non-members, post free.

In these days, when the high prices of books, particularly of those on technical and scientific subjects, show no sign of a downward tendency, the Society of Chemical Industry is to be congratulated on its ability to issue the "Annual Reports" at the very moderate charge demanded, the volume representing excellent value for the money, even at the price asked of non-members of the Society. That the "Reports" should be in the hands of every chemist is beyond question, and if a poll of the members of the Society were taken, objectors to the inclusion of the price in the annual subscription would assuredly form only a small minority.

Vol. V. follows, in the main, the paths laid down by its predecessors, the twenty-seven sections into which it is divided corresponding approximately with the headings under which the abstracts published in the Society's journal are grouped.

In view of the high cost of fuel now prevailing, and of the consequent necessity for economy in its consumption, the widest appeal to those engaged in industry is made by the first few sections, in which such matters as fuel in general, boiler plant, steam production, liquid and motor fuels, power alcohol (referred to also in several of the later sections), and the like, are considered. The section of greatest interest to the members of our own Society is that under the title "Analytical Chemistry," now for the second time from the experienced hands of Mr. C. A. Mitchell. This section deals principally with such analytical methods as are more especially of technical interest, and may usefully be read in conjunction with the chapter—also due to Mr. Mitchell—under the same title in the "Annual Report of the Progress of Chemistry for 1920," issued by the Chemical Society. Analytical processes are given a place also in certain of the other sections.

In general, the contributors have done their work thoroughly and with discrimination, and although it may be felt that some of the matter dealt with deserves more than the passing notice actually bestowed on it, the rigid rationing of space necessary with a composite compilation of this character must be held responsible.

As in former years, the book is printed in a good, readable type, and few errors, typographical or otherwise, are evident. Its value is greatly enhanced by the excellent author and subject indexes.

T. H. PORR.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

OBITUARY NOTICE.

JOHN HUGHES.

JOHN HUGHES, a very old member of the Society, who died on June 4 in his seventy-seventh year, was the son of the Reverend Isaac Hughes, Vicar of Mynyddyslwyn in Monmouthshire, and was educated at the Usk Grammar School and at the well-known Agricultural College of Cirencester, where he developed a special taste for Agricultural Chemistry, and became assistant to the then Professor of Chemistry, the late Dr. Augustus Voelcker.

When Dr. Voelcker retired from his Professorship and migrated to London, to be in closer touch with his duties as consulting chemist to the Royal Agricultural Society of England, John Hughes accompanied him, and remained a member of his staff until 1872, when he became chemist in the works of Lawes' Chemical Manure Company, where he remained until 1877, in which year he was engaged by the Ceylon Planters' Association to go to Ceylon and to make a comprehensive investigation, from the chemical point of view, of the problems affecting the growth and manuring of the coffee plant. The result of his labours was embodied in an exceedingly useful report to the Association, which was published in book form in 1879.

On his return from Ceylon, while continuing to advise Lawes' Chemical Manure Company, Hughes established himself in independent consulting practice in Mark Lane, in which practice he was actively engaged, without interruption, until a few weeks before his death.

During this period of more than forty years his attention continued to be mainly devoted to the chemistry of soils and fertilisers, and he was responsible for the introduction into the manure market of a material to which—perhaps illogically from a chemical point of view—he gave the name of "Basic Superphosphate." This consisted of superphosphate in which the soluble phosphate was converted into a "reverted" or "precipitated" form by grinding into the superphosphate a sufficient quantity of lime, thus producing a very readily available form of non-acid phosphate for use on soils deficient in lime, for which ordinary superphosphate, by reason of its acidity, might be unsuitable, especially for turnips, cabbages, and other cruciferous

crops liable to the disease known as "finger and toe." The development of the supply of basic slag, and its general use as a substitute for superphosphate in such cases, however, prevented the development of the production of basic superphosphate on anything like the scale which it might have assumed under other circumstances; but it is still produced by some of the leading fertiliser manufacturers, and by reason of its more ready availability is considered to possess, apart from the question of cost, advantages over basic slag of corresponding phosphatic content.

Hughes joined the Society of Public Analysts at an early period of its history, and served on its Council from 1891 to 1894, though of recent years his attendance at the Society's meetings had become irregular. He was also amongst the earliest Fellows of the Institute of Chemistry, on the Council of which he served from 1891 to 1894 and from 1897 to 1900.

He held the appointment of Official Agricultural Analyst for the County of Hereford, and took an active interest in all matters relating to the administration of the Fertilisers and Feeding Stuffs Act.

BERNARD DYER.



ESTIMATION OF POTASSIUM IN PRESENCE OF SODIUM, MAGNESIUM, SULPHATES AND PHOSPHATES.

By H. ATKINSON, B.A., A.I.C.

(Read at the Meeting, May 4, 1921.)

THE method depends upon the relative solubilities of these salts, and the perchlorates of the metals, in methyl alcohol. Potassium perchlorate has a low solubility in this liquid, whilst the perchlorates, sulphates, and phosphates of magnesium and sodium have a sufficiently high solubility to make the separation possible.

The procedure is as follows: The mixture of salts is evaporated in a 300-c.c. beaker with perchloric acid until fumes of the acid are evolved, and to ensure complete removal of hydrochloric acid, if present, the evaporation with perchloric acid is repeated; 100 c.c. of methyl alcohol are added, and the solution is maintained at the boiling-point, with occasional stirring, for one hour. It is then allowed to stand overnight, and filtered through a Gooch crucible, the transference and washing being effected with 50 c.c. of methyl alcohol acidified with perchloric acid in the ratio of 95 alcohol : 5 perchloric acid (sp. gr. 1.12), and finally with about 20 c.c. of ethyl alcohol.

A blank experiment was made with pure potassium perchlorate, and the loss due to the solution of the salt was 0.014 grm., but when other salts are present small quantities are held out of the solution by potassium perchlorate, and the loss is thereby compensated to a certain extent.

It is evident that so long as the quantities of the radicles named are well within the limits of their solubility the determination of potassium as perchlorate is possible.

The limit of solubility of these radicles was determined by taking an excess of a mixture of magnesium sulphate and sodium phosphate (about 3 grms. each) and, following the procedure already stated, evaporating off the alcohol from the filtrate, dissolving the residue in water, diluting the solution to 250 c.c., and determining the amounts of the different radicles in aliquot portions.

The proportions in the total filtrate were: Magnesia (MgO), 0.228 grm.; sulphates (H_2SO_4), 0.54 grm.; and phosphates (P_2O_5), 0.928 grm.

The quantities of salts used in test experiments were: (a) 0.500 grm. potassium chloride, and 1 grm. sodium sulphate, $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$; and (b) 0.499 grm. potassium chloride, 0.6 grm. magnesium sulphate, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$; and 1.1 grm. sodium dihydrogen phosphate, NaH_2PO_4 .

The weight of potassium perchlorate obtained in each experiment was 0.924 grm., corresponding to: (a) 99.4 per cent. potassium chloride, and (b) 99.6 per cent. potassium chloride.

PUBLIC HEALTH LABORATORIES, CAIRO.



THE JOINT USE OF TWO INDICATORS IN THE TITRATION OF ACIDS AND BASES.

BY J. L. LIZIUS, B.Sc., A.I.C.

(*Read at the Meeting, June 1, 1921.*)

It has been found that accurate titrations can be readily obtained, by the joint use of two indicators, where colour changes denote either the approach of the end-point or that the titration has been overshot.

1. *Titration where Phenolphthalein is used as the Indicator.*—One drop of phenolphthalein (0.5 per cent.) and 3 drops of thymolphthalein (0.04 per cent.) are added to the acid solution to be titrated. The end-point is denoted by a pink colour (P_H 8.3). On the addition of a further drop of $\frac{N}{10}$ alkali, the solution becomes a violet colour, produced by the combination of the pink colour of phenolphthalein with the blue colour of thymolphthalein (P_H 10). Thus, if the end-point has been overshot by 1 drop of $\frac{N}{10}$ alkali, this will be denoted by a violet colour, and the correct titration can be obtained by back-titration with $\frac{N}{10}$ acid, 1 drop at a time until the colour of the solution changes from violet to pink.

Similarly, in the titration of alkaline solution, when phenolphthalein is used as the indicator, it is advantageous to add thymolphthalein. The approach of the end-point is denoted by the colour change violet to pink, and the acid is then added 1 drop at a time until the pink colour disappears.

2. *Titration where Methyl-red is used as the Indicator.*—One drop of methyl red (0.02 per cent.) and 3 drops of thymol-blue (0.04 per cent.) are added to the acid solution to be titrated. The end-point is denoted by an orange colour (P_H 6). On the addition of a drop of $\frac{N}{10}$ alkali the solution becomes yellow, and a further drop of alkali produces a blue-green colour (P_H 9), due to the thymol-blue.

Thus, if the end-point has been overshoot by 2 drops of $\frac{N}{10}$ alkali, this will be denoted by a blue-green colour, and the correct titration can be obtained by back-titration with $\frac{N}{10}$ acid, 1 drop at a time, until the neutral orange tint is obtained. The methyl-red-thymol-blue combination is, however, more useful where alkaline solutions are to be treated with $\frac{N}{10}$ acid. The solution is blue, and as the end-point is approached, the colour changes from blue to green. Another drop of $\frac{N}{10}$ acid causes a colour change, green to yellow, and one more drop of $\frac{N}{10}$ acid is required to obtain the neutral orange tint.

It was found that the addition of 3 drops of the auxiliary indicators had no effect whatever on the titration values.

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THE SPECTROMETRIC EXAMINATION OF CERTAIN FIXED OILS AS A MEANS OF IDENTIFICATION.

By HERMANN C. T. GARDNER.

(Read at the Meeting, June 1, 1921.)

IN view of the conflicting statements made by Allen (*Comm. Organic Analysis*, Third Edition, II., p. 26), by Lewkowitsch (*Chemical Analysis of Oils, etc.*, Second Edition, p. 113), and by Doumer and Thibaut (*Corps Gras Industrielles*), it is obvious that any spectroscopic examination of any fixed oil exhibiting absorption bands is untrustworthy if reliance be placed upon the presence, or absence, of such phenomena for diagnostic purposes; and, as several fixed oils do not yield any absorption bands at all, simple spectroscopic examination alone would appear practically valueless as a means of identification. From what has been accomplished so far the only use of the spectroscope would seem to be for the purpose of placing an oil in a particular class—a waste of time so far as analytical procedure is concerned.

The observations recorded from my investigations are not proffered as conclusive, as, obviously, repeated examinations of similar oils from dissimilar sources are requisite before a table of definite limits of absorption or the ranges of visibility of the spectra of fixed oils could be accepted as infallible. Such a table, if free from error, would doubtless assist in the speedy identification of a fixed oil because it would prove discriminative.

With the foregoing reservation, my observations tend to show that dissimilar oils exhibit dissimilar limits of visibility under similar conditions, whilst oils of the same kind show practically identical spectra as regards length—that is, in the extent of the visibility of their spectra—under like conditions. Herein lies the true value of Allen's remark, that "no oils of animal origin give definite absorption bands, the spectrum being merely obscured at the more refrangible end."

METHOD.—The instrument used was a Browning's Student's Spectroscope fitted with a $1\frac{1}{4}$ -inch dense prism and provided with an arc of 100° divided into thirds of

a degree, having a vernier and lens, thus permitting angular measurements of the position of the axis of the telescope to be made.

The readings were taken of such angular measurements by bringing the telescope into such a position that one of the cross wires in the eye-piece, when in a vertical position, coincided with the extreme edge of the visible spectrum, so that on one side was the barely visible limit of the spectrum and on the other obscurity; or in the case of an absorption band, when the band was sufficiently wide to be conveniently measured, by bringing the wire into coincidence with the extreme edges of the band alternately. If the bands were not of such a breadth as to permit of this, the wire was brought into apparent superimposition; in each instance the reading on the scale of the arc was noted.

Measured in this manner, the length of the visible spectrum of white light was found to occupy a segment of the arc, beginning at the red end at 43° and ending at the violet at 47.6° . The range of the spectrum was thus 4.6° . The position of the sodium, or D line, was experimentally ascertained to be precisely at 44.3° , corresponding to division 50 on Bunsen's scale.

The apparatus consisted of dull black metal troughs of various lengths (usually 3 or 6 inches), having a window of clear white glass at either end. The window intended for use near the slit of the spectroscope was surrounded by a collar into which the collimator-tube of the instrument could be inserted, and on the end of the trough, remote from the spectroscope, a box-like attachment was constructed to receive the source of light.

The trough was filled with the particular oil at 30° C. to 40° C., and covered with a dull black lid, and the spectroscope placed in position after the small reflecting prism had been swung aside. From the opposite end a beam of light was transmitted through the oil from a metallic filamented $\frac{1}{2}$ -watt electric bulb of the gas-filled type (50 c.p.), and the slit of the spectroscope was suitably adjusted. As the observations were made in daylight the prism of the instrument was covered with a black cloth.

Diagrams of spectra made from the results obtained have shown that both the limits of visibility and the width of an absorption band, and in some measure its intensity, bear a relation to the horizontal thickness of the oil through which white light is transmitted.

In practice, some difficulty arises in precisely determining the edge of an absorption band, because the absorption becomes less towards the edge, which is therefore not sharply defined, but shaded off to a limit at which absorption is not detectable. Small differences due to this cause, however, are not so great as the differences, in most instances, of the extent of visibility of the spectra of oils of different nature which I have examined.

It would thus appear to be of no use merely to state the width of any absorption bands appearing in the spectrum of an oil, and if the breadth of a band be measured it should be connoted with the thickness of oil through which the light passes before entering the spectroscope. In view, however, of the untrustworthy evidence afforded by such bands too much reliance should not be placed either on their width or their presence or absence.

HOMOGENEOUS LIGHT.—The effect of passing light as nearly as possible homo-

geneous through the various oils was tried by interposing a red glass screen between the source of white light and the oil.

The visibility of the spectrum given by the screen itself ranged from 43.5° in the red to 44.3° in the yellow. It thus filtered out the whole of the violet, indigo, blue, green, and most of the yellow rays from white light, and permitted light very nearly homogeneous to penetrate the oil.

As was probably to be expected, in the case of those oils the spectra of which exceeded in visibility the spectrum of the screen, the spectrum obtained was that of the screen only.

Similarly, in the case of a mixture of two different oils yielding spectra with visibilities differing in extent, the spectrum observed when white light is passed through the mixture is (excluding any absorption bands due to chromogenetic causes in either oil) that of the oil giving the spectrum of the less extensive visibility, provided the limits of visibility at both ends come within the limits of visibility of the spectrum from the oil giving the more extended spectrum. Whether this holds good for an admixture of a very small amount of one oil with another remains yet to be determined.

SPECTROMETRIC CHARACTERISTICS.

Oil.	Three Inches of Oil used.			Six Inches of Oil used.		
	Visibility of Spectrum in the Red ends at—	Visibility of Spectrum in the Blue ends at—	Approximate Extent of Visibility.	Visibility of Spectrum in the Red ends at—	Visibility of Spectrum in the Blue ends at—	Approximate Extent of Visibility.
Almond	43°	47.3°	4.3°	43°	47°	4°
Arachis	43.3°	45.6°	2.3°	43.6°	45.3°	1.7°
Castor (East Indian)	Slightly varies about 43.2°	45 to 45.3°	2 to 2.3°	Six inches not suitable for examination		
Coconut	43.28°	47.3°	4.02°	43.3°	46.6°	3.3°
Colza (2 inches of oil)	43.3°	44.6°	1.3°	Six inches not suitable for examination		
Cotton seed (English refined)	43°	46.6°	3.6°	43°	45.6°	2.6°
Linseed, raw	Slight	45°	2.0°	Slight	44.6°	1.6°
Linseed, boiled	43.3°	44.3°	1.3°	43°	44.3°	1.3°
Neatsfoot	43.3°	45.64°	2.34°	43.6°	44.34°	About 0.74 , under 1°
Olive	Nil	45.3°	2.3°	43.16°	45.16°	2°
Sesame	43.16°	45.6°	2.46°	43.3°	45°	1.7°

CONCLUSION.—From the experimental data obtained it seems evident that careful spectrometric observations afford a quick and ready means of identifying a fixed oil, at any rate in the case of those specified.

The above table indicates the spectrometric characteristics of the oils examined,

from which it will be noticed the variations in the extent of visibility are most marked at the blue end of the spectrum, the limits of visibility at the red end not greatly varying.

It will also be seen that the lengths of the visible spectra are mostly different.

DISCUSSION.

Mr. H. C. REYNARD said he would like Mr. Gardner to give some idea of the effect of the age of the seed from which the oil is extracted on the absorption spectrum, as there seemed to be more difference in the absorption spectrum of American "butter oil," prepared from fresh cottonseed, than in that of oil from the kind of seed we are accustomed to get in England. He considered that no amount of refining would give oil, from the latter seed, of so good a colour as the American oil. He feared that as a means of diagnosis of oil the method described by the author was likely to be too much affected by the method of refining and the source of the oil, although, judging by the amount of data given in the paper, probably the writer had already studied this question.

Mr. BOLTON said that, while he was not impressed by spectrometric methods as an analyst's weapon, the readings might prove useful as a manufacturer's method of sorting oils. The data given, however, were most valuable.

Mr. CHAPMAN asked for information as to what the absorption spectrum was really due. He inquired if certain constants were peculiar to certain oils, in which case the test might become of diagnostic value—apart from the other colour, which was, to some extent, accidental. He wondered if the speaker had examined tung oil.

Mr. GARDNER, in his reply, said that Mr. Reynard's point was, perhaps, the weakness of the method. A good deal of cottonseed oil was obtainable from India, the examples dealt with in the paper, however, being imported English refined oils. Undoubtedly the process of refining removed the chromogenetic principle, and colouring matters were rather a cause of absorption bands than otherwise. The colour which remained in the oil was dependent on clarifying, as would be realised on comparison with the figures given (in the charts) for neatsfoot oil. The absorption spectrum bore no relation to the constituents of the oil.



THE SEPARATION OF ALUMINIUM FROM BERYLLIUM.—PART I.

By HUBERT T. S. BRITTON, B.Sc., F.I.C.

JUDGING from the number and variety of methods of separation of aluminium from beryllium described in the literature, it may be inferred that the problem has been the source of much difficulty. The bulkiness and gelatinous nature of both the aluminium and beryllium hydroxide precipitates render the methods involving them extremely difficult to make quantitative on account of the property possessed by each to adsorb substances from solution.

The following is an epitome of the methods which have been described :

1. Digestion of hydroxides with a concentrated solution of ammonium carbonate, causing beryllium hydroxide to pass into solution. (Vauquelin, *Ann. Chim. Phys.*, 1798, 26, 155.)
2. Treating aluminium and beryllium hydroxides with a saturated solution of ammonium chloride and boiling until all free ammonia gas has been liberated, beryllium hydroxide alone becoming soluble. (Berzelius *vide* H. Rose. *Handbuch der Analytischen Chemie*, 1851, II., 61.)
3. Decomposition of either sodium or potassium hydroxide solutions of beryllium hydroxide when diluted and boiled for a short time. (Gmelin, *Pogg. Annalen*, 1840, 50, 175.)
4. Solubility of both beryllium and aluminium hydroxides in either sulphurous acid or in a concentrated solution of ammonium hydroxide saturated with sulphur dioxide. When the solution is boiled to expel sulphur dioxide aluminium hydroxide is precipitated. (Berthier, *Ann. Chim. Phys.*, 1843, [iii.], 7, 74.) A similar method has been described substituting a solution of sodium thiosulphate, being an adaptation of Chancel's method of estimating aluminium. (*Comptes rend.*, 1858, 46, 987.)
5. Precipitation of aluminium as basic sulphate by addition of zinc to a solution in sulphuric acid. (Debray, *Ann. Chim. Phys.*, 1855, [iii.], 44, 1.) This method was claimed by Scheffer (*loc. cit.*, 1859, [iii.], 56, 112) to be satisfactory, though the digestion with zinc required from two to three days.
6. A method was described by Gibbs in 1864 (*Sill. Amer. J. Sci.*, 37, 356), in which the oxides were fused with potassium hydrogen fluoride. The fused mass was treated with boiling water containing a little hydrofluoric acid to extract the beryllium as a double fluoride, which crystallized out on cooling.
7. Rössler (*Zeitsch. anal. Chem.*, 1878, 17, 148) was able to precipitate beryllium as a double ammonium phosphate by the addition of ammonium phosphate in presence of citric acid, but only when the quantity of aluminium present was small.
8. Vincent (*Bull. Soc. Chim.*, 1880, 33, 157) stated that the salts of beryllium were insoluble in excess of dimethylamine due to the precipitation of beryllium hydroxide, whereas aluminium hydroxide was soluble. C. Renz (*Ber.*, 1903, 36, 275) applied this fact, and also showed that beryllium hydroxide was precipitated by either methylamine or mono- or di-ethylamine. He gave satisfactory data obtained from an ethylamine separation.
9. Haven's method (*Chem. News.*, 1897, 76, 111) depends upon the insolubility of $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ in concentrated hydrochloric acid and ether, saturated with hydrochloric acid gas.
10. Solubility of basic beryllium acetate in chloroform. (Haber and Van Oordt, *Zeitsch. anorg. Chem.*, 1903, 40, 465.)
11. Hart (*J. Amer. Chem. Soc.*, 1895, 17, 604) suggested the use of a strong solution of sodium carbonate, which precipitated the major portion of the aluminium hydroxide. This method was later modified by Parsons and Barnes (*loc. cit.*, 1906, 28, 1589), who recommended boiling for a half minute with a 10 per cent. solution of sodium bicarbonate.
12. Fusion of BeO and Al_2O_3 with excess of sodium carbonate for two or three

hours, and then extracting the sodium aluminate with water, beryllium oxide remaining unattacked. (Wünder and Wenger, *Zeitsch. anal. Chem.*, 1912, 51, 470-3.)

13. Method depending upon the volatility of basic beryllium acetate, which Kling and Gelin (*Bull. Soc. Chim.*, 1914, 15, 205) state can be carried out by distilling the basic acetate of beryllium under reduced pressure, 19 mm. in a current of acetic acid between 160° and 170° C. for four hours, and then for another hour at 250° C. Results within 2 per cent.

Of these, methods Nos. 1, 2, 3, 4, 11 depend upon the peculiarity possessed by beryllium of forming soluble basic salts. This may be explained by the work of Hildebrand (*J. Amer. Chem. Soc.*, 1913, 35, 864), who found that the hydrogen ion concentration necessary to precipitate aluminium and beryllium hydroxides by means of sodium hydroxide, differed only by a small amount—viz., about 10^{-3} and 10^{-5} respectively. Hence, methods of separation depending upon this difference—e.g., using a solution of sodium thiosulphate, giving hydrogen ions by hydrolysis—will not be likely to be very satisfactory.

It therefore seemed that in the separations depending upon the formation of soluble basic salts of beryllium, sufficient amounts of the reagents must be taken to maintain the necessary hydrogen ion concentration to keep the beryllium hydroxide in solution; in other words, there must be limits of solubility of beryllium hydroxide in solutions of reagents for definite temperatures and concentrations. Such limits have been found in the case of ammonium carbonate, sodium carbonate, and bicarbonate. Knowing the amount of beryllia which a reagent can hold in solution and in which aluminium hydroxide is insoluble, then it would appear at first sight that a method had been found to give a complete separation, if a sufficient quantity of the reagent had been employed. If only the phenomenon of "sorption" by colloidal precipitates could be eliminated or reduced to a minimum, the above methods might be useful.

On account of this, it was thought much more desirable to try to find the conditions necessary for obtaining a quantitative separation of beryllium hydroxide in the form which can be easily washed. The precipitate of beryllium hydroxide obtained by boiling dilute sodium or potassium hydroxide solutions was found to be granular, and when examined microscopically was seen to be composed of small particles, perhaps crystalline, of a little over 1μ in diameter. In this form it could be completely washed, and as was proved by Van Bemmelen ("Die Absorption," *Gesammelte Abhandlungen über Kolloide und Absorption*, 1910, 97) does not adsorb substances from solution.

This method has been investigated by several workers. Vauquelin in 1798 (*Ann. Chim. Phys.*, 26, 155) first noticed that alkalis dissolved beryllium hydroxide, which was again thrown down on boiling. Gmelin (*Pogg. Annalen*, 1840, 50, 175) found that, if the alkali solution was not too dilute, the deposition of beryllium hydroxide, after boiling from fifteen to twenty minutes, was complete. Similar conclusions were drawn by Schaffgotsch (*Pogg. Annalen*, 1840, 50, 183), who also stated that the precipitate was an insoluble salt of potassium, from which the potassium could be removed by washing, rendering the precipitate once again soluble. Weeren (*Pogg. Annalen*, 1854, 92, 91), Hofmeister (*J. prakt. Chem.*, 1859, 76, 3), and Joy (*Sill. Amer. J. Sci.*, 1863, 36, 83-91), all obtained unsatisfactory results. Penfield and

Harper (*Amer. J. Sci.*, 1886, [iii.], **32**, 110) found the method, using sodium hydroxide, to give a good separation when the liquid was boiled for an hour at a dilution of 800 c.c., glass vessels being as suitable as those of platinum. The method was considered best by Zimmermann in 1887 (*Diss.*, Berlin; *Zeitsch. anal. Chem.*, 1888, **27**, 61-63), who stated that quantitative results were only obtainable from potassium hydroxide solutions when boiled for fifteen to twenty minutes in a platinum basin, the dilution for 0.3 grm. BeO not being allowed to exceed 300 c.c. Haber and Van Oordt (*Zeitsch. anorg. Chem.*, 1904, **38**, 377) studied the process from the point of view of the solubility of the "crystalline" hydroxide in various concentrations of sodium hydroxide solution. Some correct separations were obtained by Wünder and Chéladzé in 1911 (*Ann. Chim. analyt.*, **16**, 205), though in the one case where they did not obtain an accurate separation they attributed it to not having used sufficient potassium hydroxide to keep the aluminium hydroxide in solution. They obtained, however, in other instances correct separations when employing less alkali.

This method has proved satisfactory in the hands of several workers, but others have tried it without success. Their failure was probably due to their having employed too much alkali to redissolve the hydroxides or to having unduly prolonged the boiling. An effort has been made to establish the conditions necessary to give correct separations, which will now be described.

EXPERIMENTAL PART.

Pure beryllium sulphate ($\text{BeSO}_4 \cdot 4\text{H}_2\text{O}$) was obtained by treating commercial beryllium nitrate with excess of fairly concentrated sulphuric acid, heating until basic sulphate had dissolved, and then crystallising the salt by pouring the solution into alcohol. The crystals were washed with alcohol and recrystallised from an aqueous solution. An analysis of the beryllium sulphate gave BeO = 14.10 per cent.; SO_3 = 45.21 per cent. Calculated as $\text{BeSO}_4 \cdot 4\text{H}_2\text{O}$, BeO = 14.16 per cent.; SO_3 = 45.17 per cent.

An exact 0.5 N beryllium sulphate solution and a number of sodium hydroxide solutions, of normalities varying from 0.1 N to 6.5 N, were made up. Ten c.c. of 0.5N beryllium sulphate solution were placed in a beaker, and just sufficient sodium hydroxide solution was run in, drop by drop, to redissolve the precipitated hydroxide, this being indicated by the disappearance of opalescence. The solutions were then diluted to 500-600 c.c. and boiled for forty minutes, the volumes being maintained by occasionally adding water, after which the beryllium hydroxide was immediately filtered off by suction, and washed until no colour was obtained on dropping phenolphthalein on the precipitates. The precipitates were ignited and weighed, and the filtrates tested for presence of beryllium by acidifying with hydrochloric acid and adding ammonium chloride and ammonium hydroxide. The results given on p. 363 were obtained :

In the cases Nos. 1 to 6 the depositions were quantitative. A slight amount of beryllium hydroxide remained in solution in No. 8. A solution of 10 c.c. 0.5 N. BeSO_4 and 41.1 c.c. 1.0 N. NaOH was diluted and boiled, in order to find what deposition was obtained when the ratio of NaOH to BeO was between 8.56 to 1 and 19.76 to 1

(cf. Exp. No. 7). The deposition of beryllium hydroxide produced was quantitative; an unweighable precipitate was formed on acidifying the filtrate with hydrochloric acid, adding ammonium chloride and ammonium hydroxide, and allowing the liquid to stand for some time.

TABLE I.

10 c.c. of 0.5 N-BeSO₄ = 2.5 millimols BeSO₄.

No.	NaOH Solution used.	Number of C.c. NaOH required to Precipitate Be(OH) ₂ and Re-dissolve it.	Millimols NaOH required to keep 2.5 Millimols Be(OH) ₂ in Solution.	Total Volume of Mixture.	Mols. NaOH / Mols. Be(OH) ₂ .
				C.c.	
1	6.51 N	2.4	10.6	12.4	4.24
2	4.0 N	3.9	10.6	13.9	4.24
3	2.84 N	6.0	11.9	16.0	4.76
4	2.0 N	8.5	12.0	18.5	4.80
5	1.25 N	14.85	13.6	24.85	5.44
6	1.0 N	26.4	21.4	36.4	8.56
7	—	—	39.1	—	15.6
8	0.113 N	500 (approx.)	49.4	510.0	19.76

From Table I. it will be observed that the amount of sodium hydroxide required when dilute solutions are used is much greater than for more concentrated solutions of sodium hydroxide. Hence the use of a concentrated sodium hydroxide solution is desirable in order to keep the ratio NaOH/BeO less than 15/1, so that complete deposition will ensue.

The amount of sodium hydroxide required to keep aluminium hydroxide in solution was investigated, and fortunately the quantity required was small.

A 0.5 N-aluminium sulphate solution was made up, pure aluminium sulphate being used. Fifty c.c. of 0.5 N-aluminium sulphate were placed in a beaker and sodium hydroxide solution run in until complete re-solution was just obtained.

TABLE II.

Results : Calculated for 10 c.c. 0.5 N-Al₂(SO₄)₃ = 1.67 millimols Al(OH)₃.

No.	NaOH used.	Number of C.c. of NaOH required to Precipitate and Re-dissolve Al(OH) ₃ .	Millimols NaOH required to Dissolve Precipitated Al(OH) ₃ .	Mols. NaOH ₃ / Mols. Al(OH) ₃ .
1	1.0 N	6.90	1.90	1.14
2	6.51 N	1.05	1.84	1.10
3	5.88 N	1.17	1.88	1.12

The amount of sodium hydroxide required is, therefore, only slightly more than that required for the quantitative formation of NaAlO₂ (cf. Heyrovsky, *J. Chem.*

Soc., 1920, 117, 1013). Solutions of aluminium hydroxide (0.4259 grms. Al_2O_3) in the minimum amount of sodium hydroxide were diluted in one case to 400 c.c., in another to 600 c.c., and in a third to 800 c.c. These were boiled, but in no instance did any deposition of aluminium hydroxide occur in less than two hours, the largest amount being from the 800 c.c. dilution, which amounted to about 4 per cent. after three hours. Diluted solutions were allowed to stand overnight, and in each case slight depositions were observed.

It has been noticed that when decomposition of the aluminate solution begins, causing a deposit (in what has been described as the crystalline form) on the walls of the beaker, the decomposition thereafter becomes more rapid. Slade and Polack (*Trans. Faraday Soc.*, 1914, 10, 150) also noticed that the deposition on surfaces such as platinum, being rougher than glass, took place somewhat more quickly. It is therefore conceivable that there may be a chance of aluminium hydroxide depositing more quickly than usual on glass, on the deposited beryllium hydroxide if allowed to accumulate on the side of the beaker. In one separation, in which boiling had been continued for an hour, a slight quantity of alumina was deposited with the beryllium hydroxide, comprising about 2 per cent. of the total precipitate. This happened to be a case where beryllium hydroxide had been allowed to deposit somewhat heavily on the beaker, especially round the edge of the surface of the solution.

The amounts deposited during forty minutes' boiling in a dilution of 500 c.c. have proved negligible, as the following results of separations show :

TABLE III.

No.	Grms. BeO.		Grms. Al_2O_3 .		Total NaOH required for Precipitation and Re-solution.	
	Taken.	Found.	Taken.	Found.	c.c.	Normal.
1	0.2067	0.2065	0.0600	0.0603	9.75	5.88
2	0.1255	0.1258	0.1703	0.1700	7.45	6.51
3	0.1255	0.1259	0.4207	0.4210	11.40	6.51
4	0.0665	0.0665	0.2491	0.2490	7.05	5.88
5	0.0653	0.0658	0.2557	0.2550	6.90	5.88
6	0.0262	0.0263	0.2552	0.2554	5.80	5.88
7	0.0137	0.0135	0.2547	0.2550	4.95	5.88

These mixtures were made up by taking weighed amounts of pure beryllium sulphate and aluminium sulphate. They were dissolved in about 30 c.c. of distilled water, and a concentrated solution of sodium hydroxide added in just sufficient quantity to cause re-solution of the precipitated hydroxides. The clear solution was then diluted to approximately 500 c.c. and boiled for forty minutes, the volume being kept constant. The precipitate was filtered off and washed until free from sodium hydroxide, and finally ignited and weighed.

After the removal of the beryllium hydroxide, the filtrate and washings were in

each case boiled for about half an hour to ascertain whether either the deposition of beryllium hydroxide appeared complete, or if the aluminate solution began to decompose. In every separation there was no deposition, proving that there is a distinct break between the total decomposition of the beryllate solution and the beginning of decomposition of the aluminate solution.

The aluminium oxide was estimated by acidifying the solution with hydrochloric acid and precipitating the aluminium with ammonium chloride and ammonium hydroxide. The aluminium hydroxide precipitate was freed from any adsorbed sodium chloride by igniting the unwashed precipitate in a platinum crucible and extracting it with water containing ammonium chloride and ammonium hydroxide. When washed, the precipitate was ignited and estimated.

From the data given it will be seen that the use of the method is quite general, no matter what ratio of beryllium to aluminium may be present. It was feared that the method would not be satisfactory when the amount of alumina present was great as compared with the amount of beryllia, on account of the amount of sodium hydroxide that might be necessary. The following table is interesting, inasmuch as it shows that the amounts of sodium hydroxide required in the foregoing analyses to get the beryllium hydroxide into solution, after having deducted the amount of sodium hydroxide required by the formula NaAlO_2 , all lie within the amounts given in the preliminary experiments. In the analysis of mixture No. 7, where the amount of BeO is small and that of Al_2O_3 is large, it so happens that the ratio of mols. NaOH to mols. BeO is as 14.8 to 1, and therefore just falls within the limits for the quantitative decomposition of beryllate solutions.

TABLE IV.

No.	Millimols.						Mols. NaOH Mols. Be(OH)_2
	NaOH re- quired for Precipitation and Re-solu- tion.	Be(OH)_2	Al(OH)_3	NaOH to Dissolve both Hydroxides.	NaOH con- verted into NaAlO_2	NaOH re- quired to keep Be(OH)_2 in Solution.	
1	57.33	8.24	1.17	37.33	1.17	36.16	4.4
2	48.50	5.00	3.33	28.50	3.33	25.17	5.0
3	74.21	5.00	8.23	39.51	8.23	31.28	6.3
4	41.45	2.65	4.88	21.52	4.88	16.64	6.3
5	40.57	2.60	5.00	20.35	5.00	15.35	5.9
6	34.10	1.04	4.99	17.04	4.99	12.05	11.6
7	29.11	0.545	4.98	13.07	4.98	8.09	14.8

CONCLUSIONS.

The conditions necessary to render quantitative the method of separation of aluminium from beryllium, depending upon the hydrolytic decomposition of beryllate solutions, have been investigated. They are: 1. Solutions which contain not more than about 0.3 grms. of BeO and 0.4 grms. of Al_2O_3 should be evaporated to about 25 c.c., and brought to the temperature of the room, after which a concentrated

solution of sodium hydroxide—e.g., 6 N-NaOH—is cautiously added until the precipitated hydroxides are just dissolved. [N.B.—Addition of too much sodium hydroxide will produce a slower and incomplete deposition of beryllium hydroxide.]

2. A suitable dilution to produce a complete deposition of beryllium hydroxide after forty minutes' boiling is 500 c.c. Prolonged boiling may cause the deposition of some aluminium hydroxide.

3. The beryllium hydroxide must be filtered from the boiling solution immediately, as the aluminate solution, if allowed to stand for any length of time, will slowly decompose; and, further, there is a tendency for a little beryllium hydroxide to re-dissolve.

The author desires to express his thanks to Professor A. J. Allmand for the interest he has taken in this work.

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NOTES.

The Editor desires to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

THE COMPOSITION OF LOCUST BEAN SEEDS.

Smetham (*J. Roy. Lancs. Agr. Soc.*, 1909) gives three analyses of locust beans, and, from the composition of these samples, it is to be presumed that the whole bean is included; there is, however, on the market a product of locust beans, the analyses of which differ materially from those recorded by Smetham, as will be seen from the following percentage figures:

	A.	B.	C.
Water	13.2	12.3	12.4
Oil	1.8	0.8	1.4
Proteins	14.6	7.1	23.0
Carbohydrates	58.6	75.0	45.2
Fibre	9.0	3.3	13.4
Mineral matter	2.8	1.5	4.6
	100.0	100.0	100.0
Food units	100	95	106

A is the whole kernel; B is the split kernel, from which the husk has been removed; and C is the "refuse" left after the removal of B. There is no question that these are anything but the genuine product of the locust bean, and are sold as such for feeding cattle.

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NOTE ON COLOURING OF THE MICROSCOPE FIELD.

It frequently happens in the examination of crystals—*e.g.*, sugar in foodstuffs—mounted in the ordinary colourless media (cedar-wood oil) that a clear definition is not obtained. If, however, the microscope field be coloured with a suitable colouring matter which does not stain the crystals, a much sharper contrast between the colourless crystal and the surrounding field is obtained. As a result the shape and size of the crystal can be more clearly seen.

In practice it is found that the deep red oil obtained by an oil extraction of alkanet root gives the best results, and the method is particularly applicable to the measurement of the size of sugar crystals in products where other bodies than sugar are present.

The method of application is exactly the same as with other mounting media, the cover-glass being pressed well down without crushing the crystals.

The oil is readily obtained by extracting (in the cold) 100 grms. of finely ground dried alkanet root with about 200 c.c. of cotton-seed or cedar-wood oil for two days, with occasional stirring, and then filtering the extract through two filter papers on a Büchner filter. A further extraction of the ground root with another 100 c.c. of oil, and the addition of the filtrate from this to the first filtrate, gives a red-coloured oil of suitable intensity of colour. Other oil-soluble dyes may be used in place of the above, but alkanet is found very satisfactory. Also, cotton-seed oil is preferable to cedar-wood oil, as in the case of the latter the colour rapidly fades.

The method should be applicable to the examination of any colourless crystals insoluble in the oil.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Detection of Manioc and Rice Flours. L. Desvergnès. (*Ann. Chim. Anal.*, 1921, 3, 205-206.) Ten grms. of the flour are heated in a 200-c.c. flask beneath a reflux condenser for five minutes with 45 c.c. of alcohol (95 per cent.) and 5 c.c. of hydrochloric acid (22 per cent.); the liquid is cooled and filtered, and the colour observed. The following results were obtained with various flours: Oat, straw-yellow colour; wheat, clear rose-yellow tint; maize (yellow), bright yellow colour; manioc, cherry-red colour; barley, clear straw-yellow tint; rice, deep rose colour; rye, yellowish rose colour. With a mixture of pure wheat flour with 10 per cent. of manioc a rosy tint was obtained, but with a mixture of wheat-offal and manioc the strong yellow-brown colour produced by the wheat-offal masked the rose tint. This difficulty was obviated by adding 50 parts of petroleum spirit and 25 parts of water per 100 parts of the alcoholic solution; the upper layer of petroleum spirit then became yellow-brown, and the lower layer (dilute alcohol) a more or less pronounced rose colour.

R. G. P.

Use of Indicators to Determine the Reaction of Whey. Y. Okuda and H. F. Zoller. (*J. Ind. Eng. Chem.*, 1921, 13, 517.) Methyl-red cannot be relied upon to give an accurate indication of the reaction of whey in the region of P_H 4 to 5.3, and it is doubtful if any indicator which covers this region could be employed because of the great protein error. W. P. S.

Estimation of Dextrose, Lævulose, Sucrose and Dextrin in Admixture. A. Behre. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1921, 41, 226-230.) When a mixture of the sugars is treated with an excess of iodine and sodium hydroxide solutions, as in the method proposed by Willstatter and Schudel (*ANALYST*, 1918, 43, 416), the dextrose alone is oxidised to gluconic acid, whilst the other sugars are not affected; the amount of iodine required for the oxidation is a direct measure of the quantity of dextrose present. If the estimation be repeated after the mixture of sugars has been inverted by heating with hydrochloric acid under the usual conditions, the additional quantity of dextrose found is that derived from the sucrose, and the amount of this sugar is thus ascertained. To estimate the lævulose, the solution remaining after the oxidation of the dextrose with iodine is treated with lead acetate to precipitate iodides and gluconic acid, and the quantity of lævulose then found by determining the cupric-reducing value of the solution. The method may be applied to the estimation of dextrose and dextrin in commercial starch syrup; the increased amount of dextrose found after the sample has been inverted by heating for three hours with hydrochloric acid is that formed by the hydrolysis of the dextrin. If lævulose be also present, it is decomposed by the prolonged acid treatment, and it is necessary to remove the products of decomposition by means of lead acetate before the dextrose is estimated iodimetrically. W. P. S.

Polarimetric Estimation of Mannitol. M. J. Badreau. (*J. Pharm. Chim.*, 1921, 24, 12-19.) The slight negative rotation of mannitol in aqueous solution is changed to a pronounced positive rotation by the action of arsenious acid in alkaline solution. The value increases with the quantity of arsenious acid, and has a maximum of $\alpha_D = +46^\circ 53'$ when the ratio $\frac{As_2O_3}{mannitol} \cong 17.5$. For the estimation of mannitol in solution, 20 c.c. are treated with 30 c.c. of a solution containing 198 grms. of As_2O_3 and 132.5 grms. of Na_2CO_3 per litre, and the mixture is filtered and polarised. The amount of mannitol in the 20 c.c. is then obtained by the formula $\alpha_D = \frac{\alpha V}{Lx}$ where $\alpha_D = 46^\circ 53'$; α is the observed rotation, $V = 50$, and L = length of the tube in dm. Should the 20 c.c. contain more than 0.339 grm. of mannitol, the original solution must be diluted and examined afresh so that the ratio of As_2O_3 to mannitol may be not less than 17.5 : 1. The temperature should be between 16° and 21° C. Alcohol interferes with the estimation and must be removed. Glycerol diminishes the action of the arsenic, but may be neglected, provided that the weight of glycerol in the volume of liquid examined does not exceed the weight of arsenious acid added. If an unknown quantity of glycerol be present, its effect may be eliminated by polarimetrically examining the solution at

successively increased dilutions, when the amount of mannitol indicated will increase to a maximum and constant value which is the true content. Mannitol cannot be estimated by the polarimeter in the presence of the following "poly-ols," which are acted upon by arsenic: Quercitol, *d*- and *l*-inositol, erythritol, *d*- and *l*-arabinol, hexols and heptols; but aldoses, ketoses, saccharoses, and polysaccharoses are unaffected, and so do not interfere with the estimation. Tartaric and malic acids are acted upon, and must be removed by lead acetate and subsequent treatment with hydrogen sulphide. Mixtures containing optically active substances and mannitol may be analysed by diluting one portion with water and another with the arsenical solution; the above formula is then applied to the difference between the polarimeter readings, which gives the rotation due to mannitol under the influence of arsenic. This procedure is applied to the estimation of mannitol in manna. For the estimation in manna wine, the alcohol is removed by evaporation, the residue is diluted to its initial volume with water, treated with lead acetate, sodium carbonate added to remove the excess of lead, and the solution decolourised with animal charcoal. In the case of mushrooms, the mannitol may be estimated by repeated extraction with boiling water and treatment of the mixed extracts with lead acetate, hydrogen sulphide, and animal charcoal; two polarimetric readings are then taken, one before and one after treatment with the arsenical solution.

H. E. C.

Manna and the Extraction of Mannitol. G. Scarlata. (*Giorn. Chim. Ind. Appl.*, 1921, 3, 254-255.)—The author describes the sources of the various commercial qualities of manna, and gives the composition of the latter. In the Ruspini method of extracting mannitol from manna, this is first extracted with water, but application of this method to manna containing not less than 15 per cent. of impurities, mostly soil, did not permit of the preparation of pure mannitol in cones. This manna was, therefore, uniformly moistened with about 15 per cent. of water, and, after twenty-four hours, subjected to a high pressure; the manna molasses expressed consists of a dense, reddish syrup, with a green fluorescence, and finds several industrial applications. The pressed cakes were dissolved in the minimum quantity of hot water, acidified with oxalic acid to precipitate colloidal clay, the solution being freed from scum, leaves, bark, etc., by skimming, and left to cool in a wooden crystallising vat. The upper layer of crystallised mannitol thus formed was separated from the lower one of soil, and, after centrifuging, dissolved in hot water and defecated with alum and lime; after filtration through felt and subsequent concentration and cooling, pure and almost colourless crystals of mannitol were obtained. Dry distillation of manna *in vacuo* at as low a temperature as possible yields a dense liquid distillate, which apparently contains the mannitol anhydrides, mannide, and mannitan, these yielding mannitol on hydrolysis. This method may serve as a means of separating mannitol from manna.

T. H. P.

Estimation of Methyl Alcohol and Ethyl Alcohol in Spirits, Medicines, Cosmetics, etc., by means of the Zeiss Immersion Refractometer. W. Lange and G. Reif. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1921, 41, 216-226.)—A definite

volume of the sample is rendered slightly alkaline and distilled through an efficient fractionating column; when the temperature of the vapours reaches 90° C. the distillation is stopped, the volume of the distillate is noted, and its specific gravity is determined. The approximate alcoholic strength is found by reference to tables, and water is added to the distillate in quantity sufficient to make the alcoholic strength exactly 50 per cent. by volume. At this concentration the specific gravity at 15° C. of methyl alcohol is the same as that of ethyl alcohol, namely 0.9346. The specific gravity should be re-determined after the dilution, and a further quantity of water added, if necessary. The refraction of the solution is then determined by means of the immersion refractometer. A 50 per cent. aqueous solution of methyl alcohol gives a scale reading of 40.2°, whilst a 50 per cent. solution of ethyl alcohol gives a reading of 85.6°. Tables are given in the original showing the scale readings of 50 per cent. alcoholic solutions containing from 0 to 50 per cent. of methyl alcohol.

W. P. S.

Detection of Tartaric Acid in Wines. L. Mathieu. (*Bull. Assoc. Chim. Sucr.*, 1921, **38**, 352-354.)—About 20 c.c. of the wine are shaken with 20 c.c. of amyl alcohol; the alcoholic layer is separated and shaken with an equal volume of water; the aqueous layer is then drawn off, evaporated to dryness, the residue is dissolved in 5 c.c. of water, and the solution treated with 1 drop of calcium chloride solution (150 grms. of crystallised calcium chloride and 40 grms. of ammonium chloride per litre) and 2 c.c. of *l*-ammonium tartrate solution (20 grms. of the salt per litre of dilute alcohol). A precipitate of calcium racemate forms immediately if the wines contain not less than 0.5 gram. of free tartaric acid per litre.

W. P. S.

Detection of Saccharin in Wine. C. von der Heide and W. Lohmann. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1921, **41**, 230-236.)—Five hundred c.c. of the wine are evaporated to a volume of about 250 c.c. to remove the alcohol, the residual solution is cooled, acidified with 10 c.c. of dilute sulphuric acid, and extracted thoroughly with ether. The ethereal solution is washed with water, the greater part of the ether is then distilled off and recovered, and the remainder is removed by evaporating the solution in a porcelain basin. The dry residue thus obtained is dissolved in a small quantity of very dilute sodium hydroxide solution; the solution is heated on a water-bath, and small quantities of 5 per cent. potassium permanganate solution are added from time to time, until a persistent red coloration is obtained; the solution is then acidified, and again oxidised by the addition of permanganate solution. Sulphurous acid is now added to destroy the excess of permanganate, the solution is evaporated to about 25 c.c., cooled, and extracted with three successive quantities of 10 c.c. of ether; the united ethereal extracts are washed with water and evaporated, and the residue moistened with 3 c.c. of water and again evaporated. This treatment with water and evaporation is repeated many times during one hour. Free benzoic acid is thus removed completely. The residue obtained finally is dissolved in water and a drop of the solution is tasted; a pronounced sweet taste indicates the presence of saccharin, and this indication may be confirmed by evapor-

ating the solution, fusing the residue with sodium hydroxide, and identifying the resulting salicylic acid by the usual tests.

W. P. S.

Action of Phosgene on Ricinolein. A. Piutti and A. Curzio. (*Giorn. Chim. Ind. Appl.*, 1921, 3, 242-244.)—When a current of phosgene is passed through castor oil heated at 100° C., a trichlorocarbonic product is obtained which, when freed from excess of phosgene by repeatedly shaking with hot water until the latter fails to assume an acid reaction, is found to contain on the average 9.03 per cent. (8.96 to 9.15) of chlorine. The products obtained in the same way from olive, arachis, almond, and sesamé oils, which are free from hydroxyl groups, contain only from 0.08 to 0.18 per cent. of chlorine, and the method is suggested as a means of detecting adulteration of castor oil. To estimate the chlorine in the chlorinated oil, 0.2 to 0.5 gm. of the latter is mixed in a platinum crucible in the shape of a short test-tube, with pure dry sodium carbonate, and the crucible then filled with the carbonate and heated, at first gently and then more strongly, to incipient fusion of the lower layer. When cold, the contents of the crucible are dissolved in dilute nitric acid and the chlorine estimated volumetrically by the Volhard method.

T. H. P.

Detection of Beef Fat in Lard by Bömer's Method. Vitoux and C. F. Muttelet. (*Ann. Chim. Anal.*, 1921, 3, 208-215.)—The melting-points of the glycerides (G) from 38 samples of lard of authenticated origin, determined by the method previously described (ANALYST, 1921, 94) were as follows: From 64° to 65.2° C., 28 samples; 63° to 63.8° C., 6 samples; 62° to 62.8° C., 3 samples; 61.4° C., 1 sample with abnormal refractive index (oleorefractometer degree at 45° C., -16.2°). The differences between the melting-point of the glycerides (G) and the melting-point of the fatty acids (A) were as follows: From 5° to 7.5° C., 31 samples; 4.8° C., 6 samples; and 4.6° C., 1 sample. In the case of five samples of beef fat the melting-points of the glycerides (G) varied from 54° to 58° C., and ($G - A$) varied from 2.0 to 4.0° C. It is suggested that the value $2G - A$ should be employed as a basis of comparison of results rather than that recommended by Bömer, viz.: $G + 2(G - A)$. In the case of the samples of lard examined, $2G - A$ was equal to or greater than 68° C., whilst for the five samples of beef fat it varied from 59.6° to 62.6° C.

R. G. P.

Sheep-foot and Ox-foot Oils. A. Bruno. (*Ann. Falsific.*, 1921, 14, 137-139.) Specimens of the oils prepared in the laboratory had the following physical and chemical characters: *Sheep-foot Oil*—Sp. gr. at 15° C., 0.917; $[n]_D^{20}$ C., 1.46805; iodine value, 84.0; saponification value, 194; and free fatty acids (as oleic acid), 0.34 per cent. *Ox-foot Oil*—Sp. gr. at 15° C., 0.9169; $[n]_D^{20}$ C., 1.4675; iodine value, 75.3; saponification value, 194; and free fatty acids (as oleic acid), 0.87 per cent. Both oils yielded a pink coloration with Bellier's resorcinol reagent.

W. P. S.

Calcium Content of White-of-Egg. H. Kreis and J. Studinger. (*Ann. Falsific.*, 1921, **14**, 148-151.)—The quantity of calcium in fresh white-of-egg varies considerably, the ash containing from 0.59 to 4.25 per cent. of its weight of calcium oxide. There is no appreciable increase in the calcium content when eggs are immersed in lime-water for several months, and the original weight of the eggs is not altered. The calcium content does not, therefore, afford any evidence as to whether or not an egg has been preserved in lime-water. The membrane enclosing the yolk is, however, rendered very fragile when an egg is immersed in lime-water, even for a relatively short period.

W. P. S.

Estimation of Lecithin. J. L. B. van der Marek. (*Pharm. Weekblad*, 1921, **58**, 989-992.)—In Hager's method of estimation (*Pharmazientische Praxis*) the lecithin is decomposed by boiling with nitric and sulphuric acids, and the phosphoric acid precipitated with ammonium molybdate. The precipitate is washed, suspended in water, and dissolved in $\frac{N}{2}$ alkali hydroxide solution, an excess of 5 to 6 c.c. being added, and the solution is boiled until all ammonia is expelled, and then titrated with $\frac{N}{2}$ hydrochloric acid. Each c.c. of $\frac{N}{2}$ alkali corresponds (according to Hager) with 1.268 mgrms. of P_2O_5 . In the author's experience this method is tedious and quite unreliable. The phosphoric acid may be estimated directly in the residue obtained after removal of organic matter by a mixture of nitric and sulphuric acids. The acid is first neutralised with alkali, and ammonium chloride and ammonia are added to the solution until a precipitate forms, which is then dissolved in dilute hydrochloric acid. Magnesia mixture is added, and the solution is treated with ammonia at boiling temperature. It is not possible, however, to determine the amount of lecithin in a sample from its phosphoric anhydride content, as the formula $C_{42}H_{84}NPO_9$ is doubtful.

W. J. W.

Volatile Oil from the Leaves of *Ocimum Gratissimum*, Linn. O. D. Roberts. (*J. Soc. Chem. Ind.*, 1921, **40**, 164-165T.)—The leaves of this plant yield on distillation about 0.1 per cent. of volatile oil, having an odour similar to that of cloves, and the following constants: Sp. gr. (15°/15° C.), 0.995-0.996, n_D^{20} , -12.7° to -14.0°, $[\alpha]_D^{20}$, -1.526-1.532. The approximate composition of the oils is: Terpenes (chiefly ocimene), 16.0; phenols (eugenol), 55.0; phenol ethers (as methyl chavicol), 5.6; alcohols (probably linalool), 13.0; esters (as $C_{10}H_{17}OH$), 0.6; and residue, 9.8 per cent. The volatile oil from *Ocimum basilicum* appears to be somewhat similar to that described above, having: Sp. gr. (26° C.), 0.890-0.940, n_D^{20} , -11.25° to -18°, and containing 30 to 46 per cent. eugenol, and the terpene ocimene.

H. E. C.

Estimation of Glycyrrhizin in Liquorice Root. P. A. Houseman. (*Amer. J. Pharm.*, 1921, **93**, 481-495.)—The author discusses the conclusions arrived at by Linz in his experimental examination of twenty-seven methods for the estimation of glycyrrhizin in liquorice, and also criticises the method devised by Linz (*Arch. der Pharm.*, 1916, **254**, 65 and 204). A detailed description is given of experimental work upon which the following method was based: The root is ground to pass a 20-mesh sieve, and dried at 50° C. to a moisture content of less than 2 per

cent. Three grms. of the powder are treated in an extraction apparatus with 50 c.c. of ether, dried, and transferred to a 100-c.c. centrifuge tube, to which 75 c.c. of 75 per cent. alcohol are added. After frequent stirring, followed by standing overnight, the tube is whirled in the centrifuge, the clear liquid is poured off, and the alcoholic extraction of the residue twice repeated. The mixed extracts are evaporated to dryness, dissolved in 10 c.c. of water, filtered, diluted to 20 c.c., and precipitated by the addition of 3 c.c. of 10 per cent. sulphuric acid. The residue is washed with two 5-c.c. portions of ice-cold water saturated with ether, and dissolved in 30 c.c. of warm 95 per cent. alcohol. The acid solution and washings are mixed and neutralised by the addition of ammonia, evaporated to about 5 c.c., and treated with 2 c.c. of 10 per cent. sulphuric acid, and the precipitate washed with iced ether-water as above. This precipitate is dissolved in 10 c.c. of warm 95 per cent. alcohol, filtered, and added to the alcoholic solution of the first residue. To the mixed solutions 2 drops of 5 per cent. ammonia are added, the solution evaporated to dryness, and the residue dried at 100° C. and weighed. The method yields results considerably higher than do previous methods, and the glycyrrhizin obtained is light in colour, intensely sweet, and free from resins and bitter substances. The method adopted by the author for the estimation of glycyrrhizin in liquorice extract is similar to the above. The estimations, which may be made upon liquorice root, should include moisture, total ash, ash insoluble in hydrochloric acid, resins, glycyrrhizin, sugars, crude fibre, and the percentage of material passing sieves of standard mesh.

T. J. W.

Estimation of Yohimbine in Yohimba Bark. A. Schomer. (*Pharm. Zentr.-h.*, 1921, 62, 169-171; *J. Chem. Soc.*, 1921, 120, ii., 360.)—Fifteen grms. of powdered bark and 150 grms. of ether are shaken together for ten minutes, 10 grms. of sodium hydroxide solution (15 per cent.) are then added, and after shaking and allowing to stand for one hour, 100 grms. of the ethereal solution are shaken and extracted with successive quantities of hydrochloric acid (1 per cent.). The acid extract is shaken with chloroform (25 c.c.), and, after separation of chloroform, is made alkaline with sodium carbonate and extracted with chloroform. The crude alkaloidal residue left on evaporating the chloroform is dissolved in a small quantity of alcohol containing a few drops of hydrochloric acid, the alcohol is evaporated, 5 c.c. of ether are added, and also evaporated. The residue is then warmed with 50 drops of alcohol, the mixture evaporated almost to dryness, and the crystalline residue treated with 50 grms. of chloroform and cooled to 0° C. The crystals are collected, dried at 100° C., and weighed.

R. G. P.

Apomorphine Hydrochloride. D. B. Dott. (*Pharm. J.*, 1921, 107, 102.)—Examination of various samples of this salt showed the water of crystallisation to lie between one-half and one mol. per mol. of the anhydrous salt. By drying nine samples in air and determining the moisture immediately the salt was observed to be apparently dry, the mean result obtained was 4.27 per cent., corresponding to three mols. of water combined with four mols. of the salt. Apomorphine hydrochloride gradually becomes anhydrous over sulphuric acid in a desiccator, and more

rapidly when heated on a water-bath. A slight loss of hydrochloric acid occurs on heating the compound at 100° C. for a protracted period, and at 140° C. a further loss occurs, together with slight oxidation.

T. J. W.

Assay of Aconite. A. R. L. Dohme. (*Amer. J. Pharm.*, 1921, **93**, 426-429.)—

The mixed ether-soluble alkaloids obtained in the chemical examination of aconite and its preparations consist of aconitine, benzoyl-aconine and aconine; the two latter do not possess the therapeutical properties of aconitine, and as it is not possible to separate them quantitatively by chemical means in such a way that the amount of aconitine may be estimated, the chemical method of analysis is useless for determining the quality of aconite. Aconitine is about 300 times as toxic as benzoyl-aconine and 3,000 times as toxic as aconine, and a physiological method proposed is based on this fact. In the case of the fluid extract, 1 c.c. is diluted to 10 c.c. with 50 per cent. alcohol; the smallest quantity of this solution which, when diluted to 1.5 c.c. with normal salt solution and injected into the subcutaneous tissues of a 400 grm. guinea-pig, will kill within twenty-four hours, is taken as the lethal dose. With aconitine, this dose is about 0.0000005 grm. per grm. of guinea-pig.

W. P. S.

Podophyllum Ash Standards. E. L. Newcombe, C. H. Rogers and C. W. Folkstad. (*Amer. J. Pharm.*, 1921, **93**, 429-432).—

Commercial samples of podophyllum frequently yield more than 3 per cent. of ash. Plump starchy roots and rhizomes usually yield less than 3 per cent. of ash, whilst shrivelled rhizomes and roots give between 4 and 5 per cent. The amount of ash is increased if the material is not cleaned properly. One sample of commercial podophyllum powder examined contained 8.88 per cent. of ash and 5.95 per cent. of ash insoluble in hydrochloric acid.

W. P. S.

Hydrastis (Golden Seal).—In the proposed standard (*ANALYST*, 1921, 143), for "acid-soluble ash" read "acid-insoluble ash."

Estimation of Formaldehyde and Paraformaldehyde in Tablets. N. Evers and C. M. Caines. (*Pharm. J.*, 1921, **106**, 470.)—In the method proposed the sample is heated with water to convert paraformaldehyde into formaldehyde, and the latter is then estimated colorimetrically by means of Schiff's reagent. A tablet is weighed and boiled with 200 c.c. of water under a reflux condenser for thirty minutes; after the solution has cooled, the condenser is washed down with water, and the solution is diluted to 250 c.c., and filtered. A standard solution is prepared by diluting 1 c.c. of 38 per cent. formaldehyde solution to 1 litre; a series of standards is then prepared in ten test-tubes, quantities of 0.1 c.c. to 1.0 c.c. of the formaldehyde solution being taken, and each diluted to 10 c.c. Ten c.c. of the filtered tablet solution are placed in another test-tube, and to each tube is added 2 c.c. of Schiff's reagent. The colorations obtained are compared after the lapse of three minutes. The Schiff's reagent is prepared by dissolving 0.2 grm. of magenta in 10 c.c. of water, saturating the solution with sulphur dioxide, and, after twenty-four hours, diluting it

to 200 c.c. Low results were obtained when the tablets were steam-distilled and the formaldehyde estimated iodimetrically in the distillate.

W. P. S.

Approximate Estimation of Commercial Cresol in Lysol. C. J. Jordan and F. Southerden. (*Pharm. J.*, 1921, 106, 479-480).—Products of somewhat varied composition are sold under the name "lysol," but most of them consist essentially of a strong solution of castor oil or linseed oil soap incorporated with commercial cresol. To estimate the amount of cresol present 60 grms. of the sample are acidified with 30 c.c. of dilute sulphuric acid and steam-distilled until the distillate no longer gives a blue coloration with ferric chloride. The whole distillate is shaken thoroughly, but not too violently, so as to ensure saturation of the aqueous layer, and then set aside for a few hours. The volume of the cresol layer multiplied by 1.04 and added to one-fiftieth of the volume of the aqueous layer gives the weight of cresol in the portion of the sample taken. The method yielded trustworthy results in the case of mixtures containing known amounts of cresol. It is generally agreed that "lysol" should contain 50 per cent. of cresol, but analyses of ten different brands showed that, in some cases, the cresol content was about 40 per cent., and in one sample 35 per cent.

W. P. S.

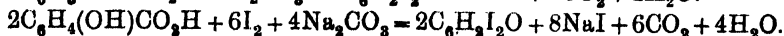
ORGANIC ANALYSIS.

Denigès' Test for the Detection and Estimation of Methyl Alcohol in the Presence of Ethyl Alcohol. R. M. Chapin. (*J. Ind. Eng. Chem.*, 1921, 13, 543-545).—Denigès's method for the detection of methyl alcohol (oxidation with permanganate and detection of the resulting formaldehyde by Schiff's reagent) is modified, as described below, in order to increase its sensitiveness and to avoid the development of false reactions. The solution to be tested is diluted until it contains 5 per cent. of total alcohols; 5 c.c. of this solution are then mixed with 0.3 c.c. of 85 per cent. phosphoric acid, and 2 c.c. of 3 per cent. potassium permanganate solution are added. When the permanganate is decomposed completely (about ten minutes), 1 c.c. of 10 per cent. oxalic solution is added, followed, after two minutes, by 1 c.c. of concentrated sulphuric acid and 5 c.c. of Schiff's reagent. If a blue or violet coloration does not appear after ten minutes (any coloration due to acetaldehyde will have disappeared in this time) there is present less than 0.2 per cent. of methyl alcohol in the total alcohol. To estimate methyl alcohol in ethyl alcohol the procedure is as follows: The solution under examination is diluted so that it contains 1 per cent. of total alcohols (solution A); 10 c.c. of this solution are mixed with 10 c.c. of 4 per cent. (by vol.) ethyl alcohol solution and diluted to 50 c.c. (solution B), and 10 c.c. of solution B are similarly mixed with 10 c.c. of 4 per cent. ethyl alcohol and diluted to 50 c.c. (solution C). Four c.c. of these three solutions are placed in separate 50 c.c. Nessler cylinders, and in a series of other cylinders are prepared a number of standards containing respectively 1, 2, and 3 c.c. of a 0.04 per cent. methyl alcohol solution; to each of the standards is added 1 c.c. of 4 per cent. ethyl alcohol and water sufficient to make the volume 4 c.c. Each of the six solutions is then treated with 1 c.c. of dilute phosphoric acid (1 vol. of 85 per cent. acid to 4 vols. of water), and 2 c.c. of 3 per cent. potassium permanganate solution; after

thirty minutes, 1 c.c. of 10 per cent. oxalic acid solution is added to each, followed, after about two minutes, by 1 c.c. of concentrated sulphuric acid and 5 c.c. of Schiff's reagent. The contents of the tubes are mixed thoroughly after each of the additions. The colorations obtained are compared after the lapse of one hour. The alcohol solution to be tested should contain alcohols only; many substances, if present, may be removed by distillation, if necessary, with the addition of alkali or acid; formaldehyde and terpenes may be eliminated by treatment with sodium hydroxide and silver nitrate, followed by distillation. Higher alcohols and acetone do not appear to interfere with the test. The Schiff's reagent is prepared by dissolving 0.2 gm. of magenta in 20 c.c. of water, adding 2 grms. of anhydrous sodium sulphite dissolved in 20 c.c. of water, then 2 c.c. of concentrated hydrochloric acid, and diluting the mixture to 200 c.c. After one hour the reagent is ready for use. W. P. S.

New Reaction of Aldehydes. R. De Fazi. (*Gazz. Chim. Ital.*, 1921, 51, I., 328-338.)—It has been already shown (ANALYST, 1916, 41, 250) that when traces of acenaphthene and of a cyclic aldehyde are dissolved in absolute alcohol, and concentrated sulphuric acid is added to the solution so as to leave the two liquids separate, the surface of contact exhibits a coloured ring, often emerald green at first, but changing, sooner or later, to reddish-violet. The same colour reactions are given if the acenaphthene be replaced by fluorene, but the reaction is not due to the formation of condensation products. T. H. P.

Estimation of Salicylates and Phenol. W. P. Emery. (*J. Ind. Eng. Chem.*, 1921, 13, 538-539.)—In a method proposed for the estimation of these compounds advantage is taken of the characteristic behaviour of the constituents, phenol and salicylic acid, towards iodine; the final product of the reaction in the presence of alkali or alkali carbonate is a purplish-red amorphous compound, $C_6H_2I_2O$, termed diiodophenylene oxide or tetraiodophenylene quinone. The reaction is represented by the equations:



Each mol. of phenol, salicylic acid, or acetylsalicylic acid yields one mol. of the iodine compound, whilst one mol. of salol yields two mols. of the same compound. The physical properties of the compound are such as to warrant its application to analysis. In the case of salol, about 0.1 gm. of the sample is weighed on to a small dry filter, and washed with successive quantities of chloroform until all soluble substances have been dissolved; the chloroform solution is evaporated at the ordinary temperature in a conical flask, the dry residue obtained is treated with 10 c.c. of 1 per cent. sodium hydroxide solution, and the mixture heated under a reflux apparatus, so that boiling begins in about two minutes. Successive quantities of 10, 30, and 50 c.c. of water are then added, the heating being so regulated that the mixture begins to boil in about three, five, and ten minute intervals respectively. Just before the last addition of water, 1 gm. of dry sodium carbonate is introduced into the top of the condenser, and washed down with the water. To the clear

boiling solution are now added 60 c.c. (or an excess) of $\frac{N}{K}$ iodine solution, the mixture again boiled, the condenser then rinsed with a small quantity of water, and the flask disconnected. A further 1 gram. of sodium carbonate is added, and the mixture boiled gently for twenty minutes; care must be taken during this period, as the evolution of carbon dioxide causes much frothing. The precipitate is then collected on a weighed filter, washed with not less than 200 c.c. of hot water, dried at 100° C., and weighed. The weight found is multiplied by 0.3113 to obtain the amount of salol present. Acetanilide, phenacetin, and caffeine do not interfere with the estimation, but when phenacetin is known to be present, the quantity of iodine solution mentioned above should be increased by 5 c.c. for each 0.1 gram. of phenacetin. With a mixture of salol and acetanilide, the filtrate from the precipitate is practically colourless; but when phenacetin is present the filtrate is coloured light yellow, so that it is not easy to recognise whether or not an excess of iodine is present.

W. P. S.

Quantitative Test of the Stability towards Heat of Nitroglycerin Explosives. M. Taliani. (*Gazz. Chim. Ital.*, 1921, 51, I., 184-193.)—A glass tube to contain the explosive is fitted with a ground cap, which is furnished with a tap, and is surmounted by a siphon tube half-filled with paraffin wax, and connected with a mercury manometer. The apparatus, with the exception of the manometer, is contained in a double copper vessel, which is closed by a double glass door, and in the jacket of which is a calcium chloride solution, with the boiling-point 120° C. for nitrated glycerine explosives, or 135° C. for nitrocellulose. The sample of the explosive is disintegrated so as to pass through a sieve of 0.6 mm. mesh, but not through one of 0.4 mm. mesh; and 1.3 grms. is introduced into the glass tube, and the apparatus is fitted together and placed in position in the bath, which has been previously heated to 120° (or 135°) C.; the tap at the cap of the tube and also one at the top of the closed tube of the manometer are left open for thirty minutes, and are then shut. The pressure necessary to maintain constant the volume of the contents of the tube holding the explosive is measured at five-minute intervals, the results being plotted against times as abscissæ. Each type of explosive corresponds with a characteristic decomposition curve.

T. H. P.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Manganese in Commonly Grown Legumes. J. S. Jones and D. E. Bullis. (*J. Ind. Eng. Chem.*, 1921, 13, 524-525.)—The following quantities of manganese were found in various leguminous plants, etc., the results being expressed as milligrams of manganese per kilo of air-dried substance: Vetch, 17 to 53; red clover, 19 to 70; Alsike clover, 40 to 140; alfalfa, 16 to 29; field peas, 20 to 52; sweet clover, 15 to 50; white clover, 33 to 35. The relative amounts in different parts of the plants were also estimated; the results obtained were as follows, and support the theory that the primary function of the manganese is catalytic (to the oxidising enzymes), since by far the largest amounts are present in the leaves:

	Stems.	Flowers.	Leaves.	Seeds.	Pods.
Red clover	20	66	84	12	
Alfalfa	13	42	76	11	
Vetch	11	17	33	10	6
Field pea	14	21	38	11	21

W. P. S.

Detection of Dextrose in Plants. M. Bridel and R. Arnold. (*Comptes rend.*, 1921, **172**, 1434-1436.)—The biochemical method of Bourquelot and Bridel (*ANALYST*, 1920, **45**, 175) has been adapted to the detection of dextrose in plants in general. The material under examination is extracted with boiling alcohol, the alcoholic extract dissolved in water, and the solution made up to definite volume, treated with lead acetate, and filtered. The filtrate is freed from lead by means of hydrogen sulphide, and evaporated to dryness under reduced pressure at a temperature not exceeding 50° C., and the residue extracted with hydrated ethyl acetate, and subsequently several times with boiling 95 per cent. alcohol. The united alcoholic extracts containing the sugars are distilled to dryness under reduced pressure in the presence of calcium carbonate, the residue dissolved in 50 per cent. methyl alcohol, the solution filtered, and the filtrate treated with emulsin in the proportion of 0.5 gm. per 100 c.c. The mixture is kept at about 20° C., and shaken at least once a day, and the reducing sugars estimated every ten days until their quantity ceases to diminish.

Estimation of Urea, Ammonia, and Amino-Acids in Urine after Precipitation of the Ammonia. M. J. Philibert. (*J. Pharm. Chim.*, 1921, **24**, 5-12, 49-58.)—After criticising the other methods for estimating the ammonia in urine, a modification of the method of Bournigault and Bith (*Comptes. rend. Soc. Biol.*, 1914, **1**, 114) is adopted which admits of the rapid estimation of ammonia, urea, and amino-acids, in the sample. The ammonia is precipitated as magnesium ammonium phosphate by adding to 40 c.c. of the urine 11 c.c. of a solution containing 100 grms. of monocalcium phosphate, and 10 c.c. of phosphoric acid per litre, 2 drops of phenolphthalein, and then 3 or 4 grms. of magnesia (free from carbonate), until, after mixing, the liquid is of a faint pink colour (excess of alkali must be avoided). The liquid is filtered with the aid of suction, the filtrate is kept apart, and the precipitate washed rapidly with 50 c.c. of water. The ammonia in the precipitate is determined by direct titration or by distillation under prescribed conditions; a correction of 25 mgrms. or 0.3 c.c. $\frac{N}{10}$ solution is allowed for the solubility of the magnesium ammonium phosphate. For the estimation of the urea 2 c.c. of lead sub-acetate solution are added to 12.5 c.c. of the filtrate from the above determination, the liquid is made up to 50 c.c., filtered, and 10 c.c. (= 2 c.c. of the urine) treated by the hypobromite process (*cf. ANALYST*, 1919, **44**, 240). The filtrate from the ammonia precipitation is also used for the estimation of the amino-nitrogen by a modification.

of the formaldehyde process. A standard colour is prepared by adding to 25 c.c. of water (coloured to match urine by the addition of a few drops of tropæolin O or OO), 2 drops of phenolphthalein, then $\frac{N}{10}$ sodium hydroxide until a faint pink tint is obtained, after which 10 c.c. of neutralised formaldehyde solution 1 : 2 are added, and finally 0.3 c.c. of $\frac{N}{10}$ alkali. For the estimation 25 c.c. of the filtrate (= 20 c.c. of the urine) are acidified with dilute sulphuric acid, phenolphthalein added, and the liquid brought to the faint pink colour, treated with 10 c.c. of the formalin solution, and titrated with $\frac{N}{10}$ sodium hydroxide until the red colour equal to that of the standard is obtained. From the number of c.c. of standard alkali required the amino-nitrogen is calculated, after deduction of 0.3 c.c. for the solubility of the magnesium ammonium phosphate, and 0.3 c.c. for the excess of alkali required to produce the standard red colour. Details as to the colour changes of the indicators and possible sources of error are discussed at length.

H. E. C.

WATER ANALYSIS.

Colorimetric Method for the Estimation of Hydrogen-Ion Concentration in Water. I. M. Kolthoff. (*Pharm. Weekblad*, 1921, 58, 1005-1020.)—For the estimation of its hydrogen-ion concentration the water is treated with neutral red solution, and the tint is compared with standardised acid and alkaline solutions of the same indicator contained in two wedge-shaped tubes attached to each other by Canada balsam. By means of a movable graduated scale the value of P_H for the corresponding tint can be read directly. In water analysis, a knowledge of the hydrogen-ion concentration is chiefly of importance in determining the carbon dioxide content when the bicarbonate content has been found.

$$[CO_2] = [H^+][HCO_3]/3 \times 10^{-7}.$$

The use of the hydrogen electrode for estimating the hydrogen-ion concentration in water is not recommended.

W. J. W.

AGRICULTURAL ANALYSIS.

Methods of Estimating the Amount of Colloidal Material in Soils. C. J. Moore, W. H. Fry, and H. E. Middleton. (*J. Ind. Eng. Chem.*, 1921, 13, 527-530.)—Large quantities of soil colloids were obtained by shaking 25-lb. portions of the soil with 125 lb. of water, separating the liquid, and passing it through a Sharples centrifugal machine; the opalescent liquid issuing from the machine was then filtered through batteries of Pasteur-Chamberlain filter-tubes, and the slimy, sticky mass collecting on the tubes was removed, stirred with water, and collected on clean filter-tubes. This material, to which the name "ultra-clay" has been given, consists mainly of hydrated aluminium silicate, and has every appearance of being a true colloid. It absorbs ammonia or dyestuffs readily, but loses this property after being heated at 1130° C.; the absorption is the same whether the material is in the form of compressed pellets or loose, incoherent powder. To estimate the colloidal matter the "ultra-clay" was dried at 110° C. for twenty-four hours, then placed in a U-tube immersed in boiling water, and the air exhausted from the tube. The U-tube was

now cooled in an ice-bath and dry ammonia gas passed through it. The absorbed ammonia was subsequently estimated by heating the tube and collecting the expelled ammonia in boric acid solution, and titrating the latter with $\frac{N}{10}$ sulphuric acid, methyl orange being used as indicator. From 93 to 110 c.c. of ammonia were absorbed per c.c. of two specimens of "ultra-clay" examined (the volume of the latter was calculated from the specific gravity). A sample of soil from which the "ultra-clay" was obtained (the specimen absorbing 110 c.c. of NH_3) absorbed 27.7 c.c. of ammonia per c.c. of soil under the same conditions, and 1.4 c.c. after it had been heated at 1130°C . Deducting the 1.4 c.c. of ammonia absorbed by non-colloidal material from 27.7 c.c., left 26.3 c.c. absorbed by the soil colloids; therefore the colloidal content of the soil was 28.9 per cent. Another method of estimating the colloidal content of the soil was based on the absorption of malachite green from a solution containing an excess of sodium oxalate; this method yielded results identical with those obtained by the ammonia absorption method.

W. P. S.

Estimation of Dicyanodiamide and of Urea in Fertilisers. E. Johnson. (*J. Ind. Eng. Chem.*, 1921, 13, 533-535.)—A volumetric method described for the estimation of dicyanodiamide depends on the formation of the complex, silver picrate dicyanoguanidine. For material containing from 5 to 15 per cent. of dicyanodiamide nitrogen, a 5-grm. portion is shaken for three hours with 450 c.c. of water and 5 c.c. of glacial acetic acid, and the mixture is then diluted to 500 c.c. and filtered. To 100 c.c. of the filtrate are added 5 c.c. of 20 per cent. nitric acid and 20 c.c. of sodium picrate solution, the mixture is cooled to 5°C ., and silver nitrate solution (7.584 grms. per litre) is added, drop by drop, until a slight excess (about 2 c.c.) over what is required for the dicyanodiamide, assumed to be present, has been introduced. After fifteen minutes the mixture is diluted to 200 c.c. and filtered, 5 c.c. of nitric acid and 3 c.c. of 5 per cent. ferric sulphate solution are added, and the excess of silver is titrated with thiocyanate solution. If 5 grms. of sample were taken, each c.c. of the silver nitrate solution used for the precipitation is equivalent to 1 per cent. of dicyanodiamide nitrogen. The sodium picrate solution is prepared by neutralising 7.5 grms. of picric acid with sodium carbonate and diluting the solution to 100 c.c.; it must be used at about 40°C . to avoid crystallisation of the sodium picrate. A method for the estimation of urea is based on the fact that this compound forms, with oxalic acid, a salt soluble with difficulty. Five grms. of the fertiliser are dried and shaken with 100 c.c. of amyl alcohol, the mixture is filtered, 50 c.c. of the filtrate are mixed with an equal volume of ether, and the urea is precipitated by the addition of 25 c.c. of a 10 per cent. solution of anhydrous oxalic acid in amyl alcohol. After thirty minutes the precipitate of urea oxalate is collected on a filter, washed twice with a mixture of equal volumes of amyl alcohol and ether, then once with ether, dried under reduced pressure, and weighed. The precipitate contains 57.01 per cent. of urea.

W. P. S.

INORGANIC ANALYSIS.

Estimation of Hydrogen-Ion Concentration in Small Volumes of Liquid by Means of Indicator Papers. J. M. Kolthoff. (*Pharm. Weekblad*, 1921, 58, 961-970.)—It has been shown that indicator papers are not reliable with very dilute solutions of acids and bases (Kolthoff, *Pharm. Weekblad*, 1919, 56, 175); on the other hand, in presence of a buffer mixture, the results agree with those obtained with indicator solutions, provided certain conditions are observed. The indicator paper should be prepared from hardened filter-paper. The drop should be applied by means of a marked capillary tube to insure uniformity of volume in each case, and the colour should be noted after a given interval and before the drop has dried. From investigation of a number of indicator papers, details of the preparation and use of which are given, the following values of P_{H+} , for the estimation of which the various indicators are suitable, are given: Congo red, 2.5-4.0; methyl orange, 2.6-4.0; blue lacmoid, 4.8-6.0; alizarin, 4.6-5.8; metachrome red, 6.0-8.5; brilliant yellow, 6.8-8.0; red litmus, 6.6-8.0; blue litmus, 6.0-7.8; azolitmin, 6.0-8.0; α -naphtholphthalein, 8.2-9.5; and turmeric, 7.5-9.5. Methyl red, phenolsulphophthalein, rosolic acid, and neutral red were found to be unsuitable. Hæmatoxylin paper may be used with strong acids; it gives a salmon colour with $\frac{N}{2}$ —, and a rose colour with 3 to 4 *N* hydrochloric acid. W. J. W.

New Reagent for Ammonia. C. D. Zenghelis. (*Comptes rend.*, 1921 173, 153-155.)—This reagent, which consists of 20 per cent. silver nitrate solution mixed with three volumes of commercial formaldehyde (33 or 37 per cent.), should be prepared shortly before being used. The solution to be tested is rendered strongly alkaline by addition of sodium hydroxide or carbonate, and 0.5 to 1 c.c. thereof placed in a test-tube about 12 cm. in height, covered by a watch-glass, from which hangs a drop of the reagent; a little water is placed in the watch-glass to serve as a condenser, the tube being then heated on a sand-bath until condensed drops collect round the reagent. With as little as 1 c.c. of 0.00002 *N*-ammonia, containing 0.00034 mgrm. of ammonia, a distinct annular silver mirror forms in the course of a few seconds. The same quantity of ammonia dissolved in a large quantity of water is detectable after the liquid has been first evaporated with a drop of dilute sulphuric acid to a volume of 0.5 to 1 c.c. The reagent will reveal the presence of ammonia in drinking water with which Nessler's reagent fails or gives doubtful results.

T. H. P.

Estimation of Silver and its Separation from Lead and other Metals by Means of Hypophosphorous Acid. L. Möser and T. Kittl. (*Zeitsch. anal. Chem.*, 1921, 60, 145-161.)—When a hot dilute solution of a silver salt is treated with an excess of hypophosphorous acid (100 per cent. more than is required by theory), the silver is precipitated completely; further heating causes the finely divided silver to coagulate so that it may be collected on a filter, washed with hot water, ignited, and weighed. Contrary to the statement by Mawrow and Mollow (*ANALYST*, 1909, 34, 115), the authors find that washing with hot water does not

cause the precipitate to pass through the filter. The method may be applied to the separation of silver from lead, cadmium, zinc, etc., since the hypophosphites of these metals are soluble in hot water.

W. P. S.

Volumetric Estimation of Mercury. E. Billmann and K. Thaulow. (*Bull. Soc. Chim.*, 1921, **29**, 587-592.)—I. Allyl alcohol reacts with mercuric compounds to form a complex organo-mercuric compound: $C_3H_5.OH + HgX_2 = (C_3H_5O)HgX + HX$. On treatment with sodium hydroxide this compound is converted into a hydroxide which is so feebly basic that phenolphthalein is not turned red until all the complex salt is converted thus: $(C_3H_5O)HgX + NaOH = (C_3H_5O)HgOH + NaX$. On treating the hydroxide with potassium bromide, quantitative liberation of potassium hydroxide occurs— $(C_3H_5O)HgOH + KBr = (C_3H_5O)HgBr + KOH$. Consequently, titration of the liquid with standard acid enables the mercury present to be estimated. From 2 to 3 c.c. of allyl alcohol are added to the mercury salt dissolved in nitric or sulphuric acid (about 5-N); the solution is then made alkaline to phenolphthalein with sodium hydroxide (2-N), and neutralised with sulphuric acid (0.2-N). A solution containing 5 grms. of neutral potassium bromide is then added, and the potassium hydroxide liberated is titrated in the usual way with standard acid.

II. An iodimetric method is based on the reaction— $HgO + KI + H_2O = HgI_2 + 2KOH$. For the estimation of mercury in salts such as the nitrate or sulphate, the salt is dissolved in an excess of the dilute acid present as radicle in the salt, the liquid is rendered alkaline to phenolphthalein with sodium hydroxide, and neutralised with very dilute sulphuric acid (the colour change is not masked by the precipitated yellow mercuric oxide); potassium iodide is then added, and the liquid thoroughly mixed in a corked flask, and finally the potassium hydroxide liberated is titrated with standard acid. The results obtained with known amounts of mercury agreed closely with those obtained by the first process and with the theoretical values.

R. G. P.

Estimation of Small Amounts of Lead in Brass. F. W. Glaze. (*J. Ind. Eng. Chem.*, 1921, **13**, 553-554.)—A method suitable for the estimation of quantities of lead of less than 0.06 per cent. in brass consists in dissolving 8.643 grms. of the sample in 30 c.c. of dilute (1 : 1) nitric acid, complete solution being effected by adding 15 c.c. of concentrated nitric acid and warming the mixture. The solution is then evaporated until cupric nitrate crystallises out, cooled, 5 c.c. of nitric acid and a small quantity of water are added, and the mixture is heated until all crystals have dissolved, and then diluted to 100 c.c. and electrolysed. Cylindrical gauze electrodes and a current of N.D.₁₀₀ of 1.5 ampères and 3 volts are employed. After about one hour the electrolyte is drawn off by means of a siphon, and the anode is washed with distilled water and alcohol, dried at 210° C. for thirty minutes, cooled, and weighed. The electrode is then cleaned, dried, and again weighed, the difference between the weights giving the weight of the lead dioxide; this weight multiplied by ten gives the percentage of lead in the brass.

W. P. S.

[This method has been used for many years in a well-known metallurgical laboratory.—EDITOR.]

Estimation of Nitrates in Bismuth Salts by means of Titanium Trichloride and Devarda's Alloy. T. McLachlan. (*Pharm. J.*, 1921, 106, 477-478.)

—Reduction of the nitrate to ammonia by means of titanium trichloride as described by Knecht (*ANALYST*, 1915, 40, 172) is best carried out in approximately neutral solution, a small excess of alkali being added before the ammonia is distilled; if a large excess of alkali was present at the beginning, the results obtained are sometimes much too low. The following procedure is recommended when the reduction is made with Devarda's alloy (copper, 50, zinc, 5, aluminium, 45 parts): Five grms. of the bismuth salt are mixed with 150 c.c. of water, 5 c.c. of alcohol, and 50 c.c. of 33 per cent. potassium hydroxide solution, and 8 grms. of the alloy are added. After about ten minutes the mixture is steam-distilled and the ammonia collected in a known amount of standard acid and titrated in the usual way. The method may be applied to the determination of nitrate in any bismuth salt, and yields consistent and trustworthy results.

W. P. S.

Estimation of Zinc as Ammonium Zinc Phosphate. G. Luff. (*Chem. Zeit.*, 1921, 45, 613-614.)—The precipitate of ammonium zinc phosphate is slightly soluble in water and more so in presence of ammonium chloride; in the latter case the solubility is to a great extent reduced by employing excess of diammonium phosphate as precipitating reagent.

W. J. W.

Gas Volumetric Method of Estimating Aluminium. L. Losana. (*Giorn. Chim. Ind. Appl.*, 1921, 3, 239-241.)—A 150 to 200 c.c. flask is charged with 0.2 gm. of the aluminium or aluminium alloy in fine turnings and 30 c.c. of boiled water, and closed by a two-holed stopper carrying a tapped funnel and a gas delivery tube which has a short rubber tube inserted in it and turns up at the lower end. The tube of the funnel is completely filled with water, and 15 c.c. of 20 per cent. potassium hydroxide solution placed in the funnel. The water in the flask is boiled to expel all air from the flask, the end of the delivery tube being then placed under a graduated tube filled with water and inverted in a trough of water and the flame removed; as soon as the pressure in the flask falls a little, the delivery tube is stopped by clipping its rubber insertion, the potash solution introduced into the flask, the flame immediately restored, and the clip removed when a slight pressure develops. The hydrogen liberated collects in the graduated vessel, the liquid being kept boiling for five minutes after evolution of gas ceases. The volume of the gas is read and reduced to 0° and 760 mm. pressure; 1 c.c. of hydrogen corresponds with 0.000802 gm. of aluminium. An alloy which may possibly be attacked by the boiling water is placed in a test-tube filled with water, and this suspended on a wire frame below the stopper of the flask, so that it can be made to fall into the boiling alkali solution. Any magnesium present acts similarly to the aluminium, and must be separately estimated and its amount deducted from the apparent quantity of aluminium, but the method is not invalidated by zinc, iron, or the small proportions of silicon and sodium normally present, and is especially suitable for alloys containing variable amounts of an inert element, such as copper, with approximately constant amounts of the other constituents; use may then be made of an empirical factor, in place of the theoretical

one, to obtain the percentage of aluminium. For alloys containing between 100 and 70 per cent. of aluminium the method gives good results. The measuring vessel consists suitably of a bulb of a known capacity of about 200 c.c., with a lower tube graduated from 200 to 300 c.c.

T. H. P.

Estimation of Cobalt and Nickel in Cobalt Steels. G. E. F. Lundell and J. I. Hoffman. (*J. Ind. Eng. Chem.*, 1921, 13, 540-543.)—From 2 to 4 grms. of the sample are dissolved in hydrochloric acid, oxidised with nitric acid and silica and tungstic acid, separated by filtration, the filtrate treated with sodium hydroxide solution, and any insoluble matter collected, dissolved in hydrochloric acid, and added to the main solution. The latter is evaporated, but not to complete dryness; concentrated hydrochloric acid is added, and the greater part of the iron, together with the molybdenum, is separated by extraction with ether. The residual acid solution is then heated with sulphuric acid until fumes of the latter are evolved, and, after dilution, the chromium, vanadium, and manganese are oxidised by potassium persulphate. The hot oxidised solution is poured into hot sodium hydroxide solution, the precipitate is collected, dissolved in dilute sulphuric acid with the aid of sodium hydrogen sulphite, and the copper is removed by treatment with hydrogen sulphide. After the solution has been boiled and re-oxidised, a double precipitation with ammonia serves to remove iron. The combined filtrates from the ferric hydroxide precipitates are evaporated to a volume of 100 c.c., neutralised with ammonia, and an excess of 35 c.c. of ammonia (sp. gr. 0.9) and 2 grms. of sodium hydrogen sulphite are added. The solution is diluted to 150 c.c. and electrolysed for eight hours, gauze electrodes and a current density of 0.2 to 0.3 ampère per sq. dm. being used; the cathode is then washed with cold water, dried at 100° C., and weighed. Traces of nickel and cobalt remaining in the electrolyte are recovered by boiling with an excess of ammonium persulphate, keeping the solution strongly ammoniacal in order to precipitate the manganese, filtering the mixture, and treating the filtrate with hydrogen sulphide. The precipitated sulphides are collected, washed, ignited, and weighed; the weight (usually not more than 1 mgrm.) is multiplied by the empirical factor, 0.75, and the result added to the weight of metals found on the cathode. The nickel and cobalt deposited on the cathode, and the small quantity of oxides recovered as described, are dissolved in 20 c.c. of nitric acid (sp. gr. 1.42), the solution is neutralised with ammonia, then rendered just acid with hydrochloric acid, and the nickel precipitated with dimethylglyoxime; the mixture is rendered slightly ammoniacal, and after two hours the precipitate is collected, dissolved in 20 c.c. of warm nitric acid, and re-precipitated as before. The precipitate is finally collected, washed, dried at 120° C., and weighed. The quantity of nickel present is calculated from the weight of the precipitate, and the amount of cobalt is obtained by difference. Methods are also described for the estimation of chromium, vanadium, copper, and manganese, in the same portion of steel.

W. P. S.

Estimation of Calcium and Magnesium in various Salt Mixtures. E. Canals. (*Bull. Soc. Chim.*, 1921, 29, 583-585.)—Iron and aluminium phosphates adsorb considerable quantities of calcium and magnesium salts when the latter are

present in relatively large proportions, but by adding acetic acid, drop by drop, with vigorous stirring, for several minutes and washing the iron and aluminium phosphates with boiling water containing acetic acid, all the calcium and magnesium passes into solution. (Cf. ANALYST, 1921, 46, 216.) R. G. P.

Constitution of Sodium Perborate. F. Foerster. (*Zeitsch. angew. Chem.*, 1921, 34, 354-355.)—The composition of sodium perborate is represented by the formula $\text{NaBO}_2 \cdot \text{H}_2\text{O}_2 \cdot 3\text{H}_2\text{O}$ and not by $\text{NaBO}_3 \cdot 4\text{H}_2\text{O}$. When the salt after dehydration at 50° to 55° C. is heated *in vacuo* to 120° C., it loses appreciable amounts of water, and the residue consists chiefly of $(\text{NaBO}_2)_2\text{O}_2$ with some $\text{NaBO}_2 \cdot \text{H}_2\text{O}$ and metaborate. The compounds $(\text{NaBO}_2)_2\text{O}_2$ differ in properties from NaBO_3 ; the existence of a substitution product of hydrogen peroxide, having the composition

$\text{NaOB} \begin{array}{c} \diagup \text{O} \\ | \\ \diagdown \text{O} \end{array}$, and analogous to persulphates and percarbonates, is implied.

W. J. W.

Use of "Fornitral" for the Detection and Estimation of Nitric Acid. (*Ann. Chim. Anal.*, 1921, 3, 207-208.)—"Fornitral" (prepared by Poulenc Frères) contains two mols. of formic acid attached to one mol. of endo-anilodiphenyldihydrotriazol $(\text{HCOOH})_2(\text{C}_6\text{H}_5)_3(\text{N}_4\text{C}_2\text{H})$. It is intended for use instead of nitron and is superior to the latter, as its aqueous solution is stable, whilst nitron must be used in acetic acid solution and deteriorates on keeping. The addition of 5 to 6 drops of "fornitral" in the cold to 1 c.c. of water containing 0.03 mgrm. of nitric acid gives a precipitate immediately; with 0.015 mgrm. of nitric acid a precipitate is obtained after two hours; and with 0.0075 mgrm. of nitric acid, after five hours. Precipitation is facilitated by rubbing the tube with a glass rod. The reagent is particularly suited for the estimation of nitric acid in presence of organic matter (*e.g.*, soil and plant extracts, culture media, fertilisers, effluents). The process is not affected by tartaric or citric acid, sucrose or glucose, urea, asparagin, aspartic acid, alkali phosphates, or salts of magnesium, calcium, lead or aluminium, but oxalic acid, when present in considerable quantities, causes high results. Nitrated compounds containing nitro groups capable of conversion to nitric acid by oxidation or hydrolysis can be estimated. For the estimation of nitric acid, the solution containing about 0.1 grm. of nitric acid per 100 c.c. is acidified with 10 drops of dilute sulphuric acid, heated nearly to boiling, and treated with 10 c.c. of the reagent (10 per cent.). The liquid is then cooled, placed in ice for about two hours, and filtered with the aid of suction through a Neubauer, Gooch, or (preferably) alundum crucible, and the precipitate is washed with a small quantity (12 c.c. in three washings) of cold water, dried at 100° to 110° C. for about an hour, and weighed. The weight multiplied by 0.168 gives the amount of nitric acid. R. G. P.

PHYSICAL METHODS, APPARATUS, ETC.

Determination of Refractive Indices of Oils. H. S. Simms. (*J. Ind. Eng. Chem.*, 1921, 13, 546-547.)—The oil to be tested is drawn into a thin-walled bulb tube, and the lower end of the tube is then sealed; the bulb should have

a diameter of 0.5 inch, and be filled completely with the oil. The filled bulb is then immersed in another oil of known refractive index, and contained in a wide test-tube. The bulb is now moved up and down, while the observer looks through it at a distant object. If the image rises as the bulb is raised, and sinks as the bulb is lowered, the refractive index of the oil is *less* than that of the medium (the oil in the test-tube), the bulb acting as a concave lens in air. If the bulb is filled with an oil having a greater refractive index than that of the medium, the image sinks when the bulb is raised, and rises when the bulb is lowered. By having a series of test-tubes containing oils of known, but different, refractive indices, and testing the filled bulb in each of these, the refractive index of the sample may be found within 0.0005 of the true value.

W. P. S.

Gold Cathodes for Electrolytic Work. A. B. Andrews. (*J. Ind. Eng. Chem.*, 1921, 13, 560.)—Open cylinder cathodes made of 24-carat gold were found to be quite as satisfactory as platinum cathodes for the electrolytic deposition of copper; the anode must still be made of platinum.

W. P. S.

New Apparatus for Steam Distillation. E. A. Roff. (*Pharm. J.*, 1921, 107, 28-29.)—The flask containing the substance to be distilled is placed entirely in the steam-generating vessel, which consists simply of a tin fitted with a lever-lid; two holes are provided in the lid, and these are closed by rubber stoppers. A long tube passes through one of these stoppers and extends into the water in the tin, thus acting as a safety tube, and the delivery tube from the flask passes through the other stopper. When the water in the tin is boiled, steam enters the flask through a tube, the upper end of which reaches just above the cork closing the flask, whilst its lower end extends into the liquid or other substance in the flask.

W. P. S.

Fractional Distillation with Contact-Ring Stillheads. R. Lessing. (*J. Soc. Chem. Ind.*, 1921, 40, 115-119r.)—For laboratory stillheads contact-rings are employed in which a diametrical partition extends the whole or part of the way across without being joined to the ring so as to interfere with the flow of liquid. Apart from their simplicity, these rings have advantages over other stillheads in that they present a large surface per unit of volume, and create turbulence in the currents of vapour and condensate by which "channelling" is avoided. Comparative curves are given for the distillation of a 1 : 1 mixture of benzene and toluene with various types of stillhead and with a plain stillhead of 4 cm. diam. and 140 cm. height, filled with $\frac{1}{4}$ -inch aluminium contact-rings. In the latter case almost complete separation was effected; benzene and toluene distilled over to the extent of 47 and 48 per cent. respectively, each within 1°, and only 5 per cent. of the mixture remained in the middle fraction. A very sharp curve was obtained in the fractionation of crude benzol from coke-oven gas, using a ring-filled stillhead and a short reflux condenser. The influence of the stillhead dimensions, heat insulation, and reflux condensing is indicated in four cases. In the fractional distillation of commercial fusel oil, using a stillhead of 4 cm. diam. and 140 cm. height, filled with $\frac{1}{4}$ -inch copper rings, and lagged with asbestos to within 10 cm. of

the top, the following fractions were obtained: From 78.0° to 87.4° C., 9.5 per cent. by volume, consisting of a mixture of aqueous ethyl and *n*-propyl alcohol; from 87.4° to 88.6° C., 19.5 per cent.; and from 88.6° to 89.0° C., 24.6 per cent., these fractions consisting of propyl alcohol. Water distilled over first at 89.1° C., and sharply indicated the beginning of isobutyl alcohol; the fraction between 89.0° and 89.5° C. was collected in two layers. When the last drop of water had distilled over, the curve showed a perfect right angle. The remainder of the isobutyl alcohol came over in an anhydrous condition between 89.5° and 107.2° C., after which amyl alcohol was collected in the remaining fractions. When aluminium rings were used for the distillation of the same sample, abnormal results were obtained. After the temperature had become constant at 89.1° C. it suddenly dropped, and at 79.6° C. white fumes were evolved; a sudden rise of temperature then occurred, followed by a drop and rise at 97.2° C., and again at 103.0° C. Aluminium hydroxide was found in the flask. The phenomenon is explained by the formation of aluminium butoxide to such an extent that the vapours reaching the thermometer were insufficient to keep it heated; air then diffused back, and traces of water in the mixture of water and butyl alcohol stopped the reaction. An interesting feature was the fact that, when the action occurred, the column still contained 69 per cent. of a mixture of butyl alcohol and water of constant boiling-point, partly as vapour and partly as condensate. The division between the moist and anhydrous portions of the column was an extremely sharp one, and the rings acted as a perfect seal against moisture.

W. J. W.



THE INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

PASS LIST.

EXAMINATIONS: JULY, 1921.

The following candidates have been successful in the recent examinations, and have been duly elected Associates of the Institute:

Examination in General Chemistry: Campbell, Alan Newton, B.Sc. (Lond.); Doolan, James Joseph; Evans, Benjamin Beardmore, B.Sc. (Birm.); Hand, Percy George Terry; Jenkin, John Watson; Pugh, William; Ridge, Bert Pusey; Woolf, Sidney Samuel, B.Sc. (Lond.).

Examination in Metallurgical Chemistry: Storer, George Paterson.

Examination in Organic Chemistry: Kenyon, Frank; Sheldon, Francis Joseph, A.R.C.S., B.Sc. (Lond.).

Examination in the Chemistry of Foods and Drugs, etc.: Martin, Charles William; Mooney, Paul Michael, B.Sc. (Lond.); Woodward, Miss Elsie.

Examination in Chemical Technology (Soap, etc.): Smith, David Dow. (*Coke Oven Practice, etc.*): Whitaker, John Wilfrid, B.Sc. (Lond.).



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FOOD CONTROL.

STATUTORY RULES AND ORDERS.

No. 1056, 1921.

ORDER DATED JUNE 24, 1921, MADE BY THE BOARD OF TRADE UNDER THE MINISTRY OF FOOD (CONTINUANCE) ACT, 1920 (10 AND 11 GEO. 5, c. 47), AND THE MINISTRY OF FOOD (CESSATION) ORDER, 1921, AMENDING THE MILK (ENGLAND AND WALES) ORDER, 1921, AND THE MILK (SCOTLAND) ORDER, 1921.*

1. The following clause shall be substituted for Clause 5 of the Milk (England and Wales) Order, 1921, and Clause 6 of the Milk (Scotland) Order, 1921:

“For the purposes of this Order the expression ‘milk’ shall include skimmed, separated, and butter milk, and any milk which has been submitted to any one or more of the following processes—viz., sterilisation, pasteurisation, homogenisation, or any other like process, but shall not include condensed milk.”

2. Copies of the above-mentioned Orders hereafter to be printed under the authority of His Majesty's Stationery Office shall be printed with the amendment provided for by this Order, and the Orders shall, on and after June 30, 1921, be read and take effect as hereby amended.

* * * * *

REVIEWS.

THE FORMATION OF COLLOIDS. By THE SVEDBERG (Professor of Physical Chemistry in the University of Upsala). Pp. vi+127. London: J. and A. Churchill. 1921. Price 7s. 6d. net.

This monograph is the first of a series by Dr. Svedberg on the Physics and Chemistry of Colloids. According to the author: “In the first monograph, I mean to give a survey of the processes which cause the formation of colloids—or of heterogeneous systems with a relatively large boundary surface on the whole (disperse systems according to W. Ostwald)—especially with regard to the conditions that determine the degree of subdivision of the systems formed. As the formation of gels is a change of state within the disperse system, it will not be considered in this monograph, which deals with the formation of colloids from non-colloid material.”

* Cf. ANALYST, 1921, 305.

Dr. Svedberg is thoroughly at home in this little book, for his own laboratory has long been famous for the researches on the formation of colloids, especially by electrical methods; this work in particular is well described and well illustrated. An extensive field is covered in little compass, being admirably summarised, critically considered, and clearly stated. Over two hundred references to the literature are made.

The following quotation from the Introduction will serve to indicate the skeleton ideas upon which the discussion is built:

"Colloids or disperse systems in general may be formed in two ways, differing in principle: by condensation, or by dispersion. In one case matter is brought together within a smaller boundary surface than before; in the other case the matter is dispersed so that the specific surface is increased. In many cases diverse systems are formed by a combined process of condensation and dispersion. Often on superficial observation the process may be considered, for instance, as a dispersion, while an intimate analysis of the phenomenon shows that a condensation process is inserted so as to make such a process the direct cause of the formation of the disperse system. . . . If the state of the system in the period that directly precedes the formation of the disperse system be taken into consideration, the classification of the processes of formation in condensation and dispersion will evidently be clearly defined."

After a discussion of the formation of disperse systems *in vacuo*, a very clear account is given of the various methods of electric pulverisation. Fifty-three pages are then devoted to the study of Condensation Processes in the formation of disperse systems in liquids and in solids. The author here gives a succinct account of the numerous investigations on the formation of the classical colloid, the gold sol, and of Odén's investigations on the relation between degree of dispersion and concentration (BaSO_4 sols). The final section, on dispersion processes, includes very brief treatments of (1) partial dissolution, (2) grinding, and (3) emulsification.

The subject of emulsification might well have received greater consideration, since, as Dr. Svedberg agrees, "the formation of disperse systems by emulsification is important both from a theoretical and from a practical point of view." The reversal of phases, the recently described method of intermittent emulsification, and the question of the limiting concentration of a purely diphasic emulsion (as distinct from concentrated emulsions obtained by using a third reagent), are matters clearly to be included in any general discussion on the formation of disperse systems.

The book is well written, and published in an attractive form. Very few errors have been noticed—*e.g.*, thioic acid, for thiosulphuric acid (p. 90), and $\text{Na}_2\text{O}_2\text{S}_3$ for $\text{Na}_2\text{S}_2\text{O}_3$ (p. 86). Readers will consider it strange to find the symbol J in KJ, AgJ and NaJ (p. 101) for iodides.

All students of colloids will welcome this little book, and find it most useful.

WILLIAM CLAYTON.

COKE OVEN AND BY-PRODUCT WORKS CHEMISTRY. By THOMAS BIDDULPH-SMITH.

Pp. x + 180. London: Charles Griffin and Co., Ltd. Price 21s. net.

A work bearing this title, written by one actively engaged in the industry, might reasonably be expected to be a treatise on the chemistry of coke-oven practice and

the application thereto of modern chemical and physical methods of control and examination. The author has, instead, given us a manual of testing such as might have been written by a "works chemist" who was not allowed on the works proper, but was confined to the laboratory. The examination of coal and coke is dealt with in four pages, thirteen lines only being devoted to the rather important matter of sampling. Another twenty-four pages are devoted to the examination and analysis of gases.

In dealing with the liquid products of the carbonisation of coal the treatment is much more thorough, and the physico-chemical methods of determining benzene, toluene, xylene, etc., in commercial benzole and solvent naphtha due to Colman, Northall-Laurie, Spielmann, and their co-workers are given in full, seven folding plates of the necessary graphs enabling a worker to use these methods without frequent reference to several large volumes. This important feature alone will make the work extremely useful to those chemists whose work includes the examination of such complex mixtures as commercial hydrocarbons.

It is to be regretted that the author has not dealt with calorimetry, pyrometry, and the physics of his subject generally a little more thoroughly. He has treated them so briefly that little could be gathered as to the use of some of the instruments described or the means which should be taken to secure reasonable accuracy during continued use.

Some uncertainty is shown as to the spelling of "benzole." On p. 138, Appendix I., Dr. Spielmann's translation of Weger's monograph on "Some Constituents of Coal Tar and their Properties" correctly states that the use of the German termination *ol* for both commercial and pure benzene and toluene "is objectionable as being unnecessary and also in causing confusion, as it is properly reserved for the alcohol group (phenol, not phenole)." "Benzol," however, occurs throughout the book, but in the index "benzole" only is given. The misprint *ortha* cresol occurring three times on p. 43 somewhat offends the eye.

This book, notwithstanding the shortcomings indicated, is a valuable contribution to the literature of chemical industry, more especially as there are full references to the original sources of information. It is to be hoped that in a new edition, revised in a more leisurely manner, the author may give us a work more representative of his knowledge and experience.

In addition to Appendix I. already mentioned, a second appendix contains many useful numerical and other data.

J. H. COSTE.

CRITICAL MICROSCOPY. By A. C. COLES, M.D., D.Sc. Pp. viii + 100. London: J. and A. Churchill, 1921. Price 7s. 6d. net.

The title of this work gives one a mild feeling of surprise by the thought that the small capacity of the volume should be sufficient to contain the whole of the subject indicated. Further investigation, however, shows that the contents are limited to the application of critical microscopy in the study of bacteriology and protozoology only, and some indication of this might have been given in the title or sub-title.

The first thirty-three pages describe the microscope and its various accessories, whilst the remainder of the book is devoted to manipulation, photomicrography, and one-and-a-half pages to the staining and mounting of objects.

The general style of the book is decidedly fragmentary, owing to the frequent interposition of extracts from the works of eminent microscopists, including many from the writings of E. M. Nelson, expressed in his usual unpleasantly interrogative style. So large a proportion of the subject-matter of the book is derived from the latter writer, that he is entitled to have his name coupled with that of the author upon the title-page. The information furnished, based largely upon the author's experience, is excellent, and should be of great value to any worker who, having mastered the elementary principles of microscopy, desires to obtain the best results possible from his instrument. Careful and accurate descriptions are given of methods for determining magnifying powers, optical indices, cover-glass correction, etc., and the means necessary for securing correct critical illumination is dealt with in an admirable manner. Any worker applying the principles and methods of manipulation advocated in this volume should meet with little difficulty, even when using the highest magnifications.

It is perhaps too much to expect that a book of this type should be free from errors, but such as occur are fortunately not serious. On p. 12 we are informed that "the Zeiss and Winkel lenses are . . . engraved 10 and 12, which . . . refer to the thickness of the cover-glass in millimetres," whilst on p. 72, line 9, "uncovered objective" should obviously read as "uncovered object." On p. 93, E. M. Nelson makes light of the effects of vibration during exposure in photomicrography, but the reviewer has lost many plates in the past from this cause, even when using a magnification of $\times 500$, when working in a laboratory adjoining an electrical power-house, and on other occasions in a building the foundations of which were close to the District Railway. The method advocated on p. 96 for the removal of dried balsam, cement, etc., from slides by the use of a knife is hardly one to be recommended, since the surface is liable to be scratched, and such blemishes must be very confusing when examining dry unmounted films microscopically. Information of but little value is given in reference to a micrometer for measuring the thickness of cover-glasses, etc., on p. 31, where the price is given as "pre-war 3s. 9d." The cost of this appliance at the present time is likely to be of more use. We are informed in the Preface that the author has "also given a short account of photomicrography with a very simple camera, by means of which one is able to take . . . sixteen photographs on a quarter-plate," but on referring to p. 89 for the method employed we find that "it is very difficult to describe on paper," and the only information obtainable is that a piece of sheet zinc with a hole 25 by 19 mm. cut in it is used.

The Index provided shows evidence of hurried compilation, for, whilst containing some incomplete references, such as "Back of objective," "Dust and dirt," "Indian ink method," and "Measurement," other subjects contained in the volume, including "Slides, cleaning of," "Development of plates," "Vibration in photomicrography," "Staining of objects," etc., are omitted.

The illustrations throughout the volume are good, and the twenty-seven photomicrographs of pathogenic protozoa, with magnifications ranging from $\times 400$ to

× 3,000, are excellent examples of the author's results, although seen at a disadvantage against the white background surrounding them. T. J. WARD.

A FRENCH-ENGLISH DICTIONARY FOR CHEMISTS. By A. M. PATTERSON, PH.D.
Pp. 384. New York: J. Wiley and Sons, Inc. London: Chapman and Hall.
Price 18s. net.

Some twenty years ago a German chemical society planned the publication of a complete chemical dictionary in English, French, and German, in which place was to be found for all the technical terms associated with each branch of chemical industry. Unfortunately, after an immense amount of material had been compiled, the scheme had to be abandoned for lack of funds, and the chance of an ideal chemical dictionary in the three languages was lost.

Apart from such an elaborate work as this, which could only have been produced by the collaboration of a large number of chemists, there has long been a demand for smaller dictionaries in French and German, containing most of the words which chemists need to know in order to read chemical and general scientific papers in those languages. To meet this want a concise German chemical dictionary was compiled, four years ago, by Dr. Patterson, and this has now been followed by a companion volume in French by the same author.

In each case the same plan has been adopted. In addition to the usual chemical terms, there is a general vocabulary of common words of most frequent occurrence, so that reference by a beginner to a second dictionary is largely obviated. In both dictionaries the basis and development of chemical nomenclature in the respective languages is concisely explained, so that it is usually possible to find the corresponding English equivalent, even when the actual word is not given.

Students only slightly acquainted with French chemical literature are frequently puzzled by the common use of colloquial or archaic words instead of the modern chemical terms, such as, for example, *sel de Seignette*, *sel de prunelle*, *sel diuretique*, etc. In translating these and similar terms the author of these dictionaries has usually given both the literal English equivalent and the chemical name of the compound, although there are a few exceptions to this useful rule, such as, for example, *eau de Labarraque*, and some omissions. In like manner, the composition of many pharmaceutical preparations is indicated in brackets after the translation, and the names of the various herbs associated with pharmacy are included, together with the more common medical terms.

The author must be congratulated upon having compressed so much into so small a space, and this French dictionary may be as warmly recommended to chemical students as its German predecessor.

Both books are bound in flexible covers of a size convenient for the pocket, and are clearly printed on good paper.

EDITOR.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE COLORIMETRIC METHOD OF DETERMINING HYDROGEN ION CONCENTRATION: SOME APPLICATIONS IN THE ANALYTICAL LABORATORY.

By NORMAN EVERS, B.Sc., F.I.C.

(Read at the Meeting, June 1, 1921.)

HITHERTO in the literature of analytical chemistry the terms "acidity" and "alkalinity" have in general been very loosely employed. "Acidity" or "alkalinity" have usually been determined by titration, and expressed in one of two ways: (a) As the number of c.c. of standard acid or alkali required by a given weight of the substance in order to render it neutral to some indicator; (b) as the percentage of some acid or alkali to which the acidity or alkalinity is ascribed, in some cases quite arbitrarily. If we require to find the percentage of a single acid or alkali which we know to be present, there is no objection to the titration method, but in many cases the important point is not the total percentage of acid or alkali present, but the number of hydrogen or hydroxyl ions present.

Hydrogen ion concentration rather than the amount of acid present is the important factor in the curdling of milk, the precipitation of proteins, the hydrolysis of sugars, and processes of digestion or fermentation, to take only a few instances. The majority of bacteria, yeasts, etc., have a range of hydrogen ion concentration at which their growth is at a maximum, and for this reason this factor is of the greatest importance in the case of bacteriological media and in biological processes generally. The complete precipitation of metals as their sulphides in inorganic analysis is dependent on the hydrogen ion concentration of the solution.

If "acidity" or "alkalinity" were expressed in terms of the hydrogen ion concentration of a solution of known strength, this would merely involve the use of a number without reference to any particular indicator, acid or alkali.

Owing to the work carried out in recent years the accurate determination of hydrogen ion concentration is now a comparatively simple matter. If only reasonable accuracy is required determinations can be carried out as a routine test in any laboratory not involving any special apparatus or skill.

METHOD OF EXPRESSING HYDROGEN ION CONCENTRATION.—Dr. Monier Williams ANALYST, 1921, 315) has dealt with the theoretical considerations involved. The

symbol P_H , by which hydrogen ion concentration is expressed, indicates the logarithm of the reciprocal of the concentration of hydrogen ions in terms of normal—*e.g.*, when $P_H = 2$, the hydrogen ion concentration is $N \times 10^{-2}$, or one hundredth normal; when $P_H = 3$, one thousandth normal, and so on. At the point of absolute neutrality,—*i.e.*, for pure water— $P_H = 7$. When P_H is less than 7 the liquid is acid, and when P_H is greater than 7 the liquid is alkaline.

METHODS OF DETERMINATION—(a) *The Hydrogen Electrode or Electrometric Method*.—This is the most accurate method of determining hydrogen ion concentration. It has been described by Dr. Monier Williams (*loc. cit.*). Its disadvantages are the somewhat complicated apparatus required, and the fact that its use involves difficulties where a series of determinations is to be made.

(b) *The Colorimetric or Indicator Method*.—This method, though sufficiently accurate for ordinary purposes, is not capable of as great accuracy as the electrometric method. It requires, however, no special apparatus, and a large number of determinations can be carried out at the same time. It is inapplicable to very dark solutions, though it may be used for coloured solutions if the colour is not too deep. It may be used with success with turbid liquids such as milk.

The colorimetric method depends on the fact that with every indicator the colour change extends over a characteristic zone of hydrogen ion concentration; if, therefore, the hydrogen ion concentration of an unknown liquid lies within the range of a certain indicator, we can determine the factor with accuracy if we can find a solution of known hydrogen ion concentration which gives the same shade of colour with the indicator. The two essentials for this method are, therefore: (a) A complete series of indicators with well-marked colour changes which will cover a wide range of hydrogen ion concentration. (b) Solutions of known hydrogen ion concentration which are easily prepared and stable. By the use of some of the recently synthesised indicators of the phthalein series, together with methyl red, we can fulfil the first requirement. As the accompanying chart shows, these indicators show brilliant and permanent colour changes over a range of $P_H = 1$ to $P_H = 11$. If we have a solution of unknown hydrogen ion concentration, and we test it with various indicators until one is found to give a shade of colour intermediate between its extreme shades, we can then find a solution of known hydrogen ion concentration which gives the same shade of colour with the same amount of indicator.

SOLUTIONS OF KNOWN HYDROGEN ION CONCENTRATION.—It is not possible for this purpose to use solutions of acids or alkali of known strength on account of the susceptibility of such solutions to changes of hydrogen ion concentration from accidental causes, such as the alkalinity of the glass, etc., but this difficulty may be overcome by introducing certain salts, such as phosphates, borates, etc., which have a "buffer" action—*i.e.*, which have the property, when in solution, of causing the hydrogen ion concentration to be only slightly affected by the addition of small amounts of acid or alkali. For ordinary purposes the following four solutions, together with $\frac{N}{10}$ hydrochloric acid and $\frac{N}{10}$ sodium hydroxide, are all that are required. By taking definite volumes of any of these solutions, with different volumes of $\frac{N}{10}$ hydrochloric acid or $\frac{N}{10}$ sodium hydroxide, solutions of practically any P_H required may be obtained.

INDICATORS.

P_H VALUES.

	1.0	2.0	3.0	4.0	5.0	6.0	7.0	8.0	9.0	10.0	11.0
Thymol blue ...	Pink	Orange	Yellow	—	—	—	—	Yellow	Greenish Blue	Blue	—
Brom-phenol blue	—	—	Yellow	Greenish Violet	—	—	—	—	—	—	—
Methyl red	—	—	—	Pink	Orange	Yellow	—	—	—	—	—
Brom-cresol purple	—	—	—	—	Yellow	Greenish	Purple	—	—	—	—
Brom-thymol blue	—	—	—	—	—	Yellow	Green	Blue	—	—	—
Phenol red	—	—	—	—	—	—	Yellow	Orange Red	—	—	—
Phenolphthalein ...	—	—	—	—	—	—	—	Colourless	Colourless	Pink	—
Thymolphthalein ...	—	—	—	—	—	—	—	—	—	Colourless	Blue
	$\frac{N}{10}$	$\frac{N}{10^2}$	$\frac{N}{10^3}$	$\frac{N}{10^4}$	$\frac{N}{10^5}$	$\frac{N}{10^6}$	$\frac{N}{10^7}$	$\frac{N}{10^8}$	$\frac{N}{10^9}$	$\frac{N}{10^{10}}$	$\frac{N}{10^{11}}$

Hydrogen Ion Concentration.

Neutral

Acid

Alkaline

1. *Standard $\frac{N}{10}$ Sodium Citrate Solution.*—This is prepared by dissolving 21.008 grms. of pure citric acid in 200 c.c. of *N*-sodium hydroxide solution and diluting to 1,000 c.c. with water.

0.1 <i>N</i> -Citrate Solution.	0.1 <i>N</i> -HCl.	P _H .	0.1 <i>N</i> -Citrate Solution.	0.1 <i>N</i> -NaOH.	P _H .
C.c.	C.c.		C.c.	C.c.	
1.0	9.0	1.17	9.5	0.5	5.02
2.0	8.0	1.42	9.0	1.0	5.11
3.0	7.0	1.93	8.0	2.0	5.31
3.33	6.66	2.27	7.0	3.0	5.57
4.0	6.0	2.97	6.0	4.0	5.97
4.5	5.5	3.36			
4.75	5.25	3.53			
5.0	5.0	3.69			
5.5	4.5	3.95			
6.0	4.0	4.16			
7.0	3.0	4.45			
8.0	2.0	4.65			
9.0	1.0	4.83			
9.5	0.5	4.89			
10.0	0.0	4.96			

2. *Standard $\frac{N}{5}$ Sodium Borate Solution.*—This is prepared by dissolving 12.404 grms. of pure boric acid in 100 c.c. *N*-sodium hydroxide solution and diluting to 1,000 c.c. with water.

0.2 <i>N</i> -Borate Solution.	0.1 <i>N</i> -HCl.	P _H .	0.2 <i>N</i> -Borate Solution.	0.1 <i>N</i> -NaOH.	P _H .
C.c.	C.c.		C.c.	C.c.	
5.5	4.5	7.94	9.0	1.0	9.36
5.75	4.25	8.14	8.0	2.0	9.50
6.0	4.0	8.29	7.0	3.0	9.68
6.5	3.5	8.51	6.0	4.0	9.97
7.0	3.0	8.68	5.0	5.0	11.08
7.5	2.5	8.80			
8.0	2.0	8.91			
8.5	1.5	9.01			
9.0	1.0	9.09			
9.5	0.5	9.17			
10.0	0.0	9.24			

3. *Standard (M/15) Potassium Dihydrogen Phosphate Solution* is prepared by dissolving 9.078 grms. pure potassium dihydrogen phosphate (KH_2PO_4) in 1,000 c.c. water.

4. *Standard (M/15) Sodium Phosphate Solution* is prepared by dissolving 23.87 grms. pure sodium phosphate ($\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$) in 1,000 c.c. water.

The two last solutions are used in combination.

PHOSPHATE STANDARDS.

M/15 Na_2HPO_4 Solution.	M/15 KH_2PO_4 Solution.	P_H .	M/15 Na_2HPO_4 Solution.	M/15 KH_2PO_4 Solution.	P_H .
C.c.	C.c.		C.c.	C.c.	
0.0	10.0	4.49	6.0	4.0	6.98
0.1	9.9	4.94	7.0	3.0	7.17
0.25	9.75	5.29	8.0	2.0	7.38
0.5	9.5	5.59	9.0	1.0	7.73
1.0	9.0	5.91	9.5	0.5	8.04
2.0	8.0	6.24	9.75	0.25	8.34
3.0	7.0	6.47	9.9	0.1	8.68
4.0	6.0	6.64	10.0	0.0	9.18
5.0	5.0	6.81			

The following solutions are also required :

$\frac{N}{10}$ Sodium Hydroxide Solution (Free from Carbonate).—One hundred grms. of pure sodium hydroxide are dissolved in 100 c.c. water in a flask covered with tin-foil, and allowed to stand overnight for the carbonate to settle. The solution is then filtered quickly with the aid of the pump through a hardened filter. Ten c.c. are diluted to about $\frac{N}{7}$ strength with distilled water free from carbon dioxide, standardised against potassium hydrogen phthalate to phenolphthalein, and diluted to $\frac{N}{10}$ strength. This solution is stored in a bottle coated with paraffin wax and connected by a glass tube with a burette. The bottle and the burette have side-tubes joined to soda-lime tubes to prevent the entrance of carbon dioxide.

$\frac{N}{10}$ Hydrochloric Acid.—The ordinary laboratory solution may be used. The above solutions, with the exception of the $\frac{N}{10}$ sodium hydroxide, are kept in well-stoppered resistant glass reagent bottles. The only other apparatus required consists of burettes, graduated pipettes, suitable dropping bottles for the indicators, and racks for the test-tubes.

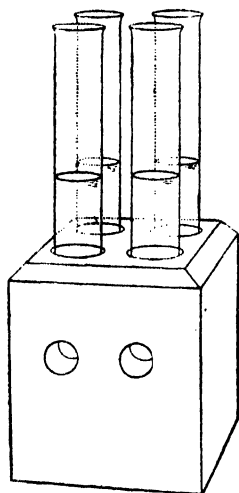
INDICATOR SOLUTIONS.—The following table shows a complete series of indicators which may be used for solutions from $\text{P}_\text{H}=1$ to $\text{P}_\text{H}=11$. These indicators are now readily obtainable on the market.

			Strength of Solution.	Range of P_H .
			Per Cent.	
Thymol blue ...	...	...	0.04 in water	{ 1.2 to 2.8 8.0 to 9.6 2.8 to 4.66 4.4 to 6.0 5.2 to 6.8 6.0 to 7.6 6.8 to 8.4 10.0 to 11.0
Brom-phenol blue ...	...	...	0.04 "	
Methyl red ...	...	...	0.02 in 50 per cent. alcohol	
Brom-cresol purple ...	...	...	0.04 in water	
Brom-thymol blue ...	...	...	0.04 "	
Phenol red ...	...	...	0.02 "	
Thymolphthalein ...	...	...	0.04 in alcohol	

METHOD OF DETERMINATION—1. For Clear or Turbid Liquids Free from Colour.

—The solution to be tested is tried with various indicators until one is found which gives a tint lying between its extremes of colour. Ten c.c. of the solution are then run into a clean test-tube, washed with neutral distilled water, and 5 or more drops of the indicator solution are added. With a little practice it is easy to judge from the shade of colour roughly what the P_H value is. Ten c.c. of a solution of this P_H value are then prepared in another test-tube from the standard solutions by consulting the tables—e.g., if the solution gives a neutral colour with brom-phenol blue, the P_H may be judged to be about 3.5. From the tables we see that a mixture of 4.75 c.c. of $\frac{N}{10}$ citrate solution and 5.25 c.c. of $\frac{N}{10}$ hydrochloric acid has a P_H value of 3.53. This mixture is therefore prepared by running in the solutions from burettes or graduated pipettes. Five drops of the indicator are added and the colours compared. If the shades of colour do not match, another tube of a different P_H value is prepared until an exact match is obtained.

2. *For Coloured Liquids.*—For this purpose the piece of apparatus known as a comparator is used (see diagram). It consists of a cubical block of wood of $3\frac{1}{2}$ inches side, with four holes bored vertically to hold four test-tubes. Two holes are also bored horizontally completely through the block, so that it is possible to look through two pairs of test-tubes simultaneously. In hole No. 1 is placed the tube containing 10 c.c. of the solution to be tested and 5 drops of indicator. Hole No. 2, behind this, holds a tube containing 10 c.c. of distilled water. In No. 3 we have the tube containing 10 c.c. of the standard solution with 5 drops of indicator, and, behind this, in No. 4 is a tube containing 10 c.c. of the coloured solution. In this way the colour is compensated, and we can compare the shades of colour without interference from the colour of the solution. If the colour of the solution is very dark, it may not be possible to distinguish the colour of the indicator, and recourse must be had to the electro-metric method. Where a large number of determinations has to be made it is convenient to have racks holding test-tubes containing a series of standard solutions with the indi-



cator, so that the tubes can be easily picked out for comparison and replaced. In this way a large number of solutions can be dealt with in a very short time. With practice it is quite easy to get a rough idea of the P_H value of a solution by merely adding the indicator without the use of standard solutions, and in many cases this may be all that is required.

THE DETERMINATION OF ACIDITY OR ALKALINITY OF COMMERCIAL ARTICLES.

—The method is very useful where a large number of samples have to be compared for acidity, such as gelatin, flour, starches, milk, sulphur, and many others. For merely comparative purposes all that may be necessary is to add the indicator to a suspension or solution of known strength and compare the colours without determining the actual P_H value.

Fine Chemicals.—Every pure salt when in a solution of definite concentration

should give a solution of definite P_H value. The method may therefore be used as an indication of the purity of salts and of the presence of an excess of the free acid or base. Where a strong acid is combined with a strong base the P_H of the solution should be about 7. If the base is stronger than the acid the P_H of the solution will be greater than 7, and if *vice versa*, the P_H will be less than 7.

The following are a few of the more interesting results obtained with commercial chemicals.

Sodium Salicylate.—Pure sodium salicylate prepared by dissolving pure salicylic acid in the calculated amount of pure sodium hydroxide solution was found experimentally to have in 2 per cent. solution $P_H=9.1$. A large number of commercial samples, on the other hand, were found in 2 per cent. solution to lie between $P_H=4.4$ and $P_H=5.8$. The reason appears to be that manufacturers are careful to keep an excess of salicylic acid present in order to avoid the discoloration which occurs if the salt is allowed to become alkaline. The excess of salicylic acid is found on titration to average about 0.1 per cent.

Morphine Hydrochloride.—The P_H of a 1 per cent. solution of morphine hydrochloride was found by experiment to be 3.65. Several commercial samples varied from 4.5 to 4.8, evidently containing a very slight excess of morphine. One sample, however, had a P_H of 6.5. It was not so readily soluble as usual, and was shown to contain free morphine.

Calcium Chloride.—A sample of crystalline calcium chloride was found to have a P_H in 2 per cent. solution of over 11. Calcium chloride, if pure, should give a practically neutral solution. The result was found to be due to the presence of free lime in the sample.

Potassium Iodide.—The reaction of this salt is of considerable importance from the pharmacist's point of view, as there must be no possibility of iodine being set free in the various combinations in which it is likely to occur. For this reason a salt with a slight alkaline reaction is to be preferred. If the P_H of a 2 per cent. solution is not less than 7 nor more than 9, the salt will be satisfactory from this point of view.

As other instances in which the method may be useful the determination of the alkalinity of natural waters and of soap solutions may be mentioned. For the standardisation of the reaction of bacteriological media it has to a very large extent replaced the old method of titration to phenolphthalein.

In the author's experience, in a commercial laboratory dealing with a large variety of materials, this method is finding fresh uses almost daily. There are probably many other analytical problems, besides those mentioned above, to which this method might be applied. The aim of this paper, for which but little novelty is claimed, is mainly to illustrate to those analysts who have not yet had experience of it what is, in the author's opinion, a valuable addition to analytical methods.

My thanks are due to Messrs. Allen and Hanburys, Ltd., in whose laboratories this work was carried out.

DISCUSSION.

Mr. W. PARTRIDGE said that the National Health Insurance Committee had given attention to the question of hydrogen ion concentration in so far as it was

applicable to bacteriological media, and the results of their investigation could now be obtained in pamphlet form. Referring to the personal element, which was so important in colour matching, he pointed out that the matching apparatus was independent of the original colour of the solution.

Mr. C. L. L. CLAREMONT questioned the method of expressing the results on the ground that it was a purely conventional way of expressing something we did not understand at all. In his opinion such methods as these did not mark any advance in general laboratory work, and he did not consider the method really useful. Many years ago, when other indicators were used, the results were quite as accurate and good as now.

Mr. B. S. EVANS said that there was considerable conventionality in the use of indicators, and that different indicators did give different results with different acids, and he considered it all to the good that some attempt should be made to express acidity, independently of the indicator. He would be glad to know if Mr. Evers knew of any apparatus for balancing extraneous colours in ordinary colorimetric work, as, for instance, in the colorimetric estimation of manganese in copper, where the colour of the iron present interferes with the matching of the permanganate. He cited the case of acid phosphates as one in which ordinary titration with indicators was useless.

Mr. HAWKINS said the chief difficulty was the question of carbon dioxide. Small quantities of carbon dioxide were liable to creep in, and everybody knew that in the case of the Kjeldahl process such small quantities made a distinct difference. He would be glad to have a little information as to an indicator sufficiently insensitive to carbon dioxide and yet acting as a sharp indicator in artificial light.

Mr. BOLTON asked if Mr. Hawkins had tried cochineal for Kjeldahl estimations, as he had found it sensitive both by day and night, and he thought it would be difficult to find a better indicator for that purpose.

Mr. CHAPMAN, after thanking the author for having brought the matter before the meeting in such a clear manner, remarked that everyone knew how unsatisfactory litmus, methyl orange, etc., were, on occasions, as indicators, and alluded, as an example, to the adjustment of the hydrogen ion concentration of soluble starch used in Lintner's method of determining diastatic activity. With these new indicators one could, at any rate, get definite results.

Mr. EVERS, in reply, stated that he had had considerable experience with this method and the methods in general use, and had obtained far more uniform results in this way than by the ordinary method of standardisation. Hydrogen ion concentration could be determined rapidly—rapidity was its great point, and the trouble of titration was eliminated. It was a means of getting something absolutely definite; you did understand what you were determining. So far as he knew, there was no previously described apparatus suitable for Mr. Evans's purpose, but probably the apparatus he had demonstrated would be suitable. He had always used brom-phenol blue for ammonia titrations, as he had found that it was not interfered with by carbon dioxide. He did not consider that cochineal had any advantages over these indicators, and it was not so effective for determining hydrogen ion concentration.

GEMSBOK BEANS.

(Bauhinia esculenta Burch.)

By G. T. BRAY, A.I.C.

"GEMSBOK" beans, derived from *Bauhinia esculenta* Burch. (N.O. *Leguminosæ*), a plant indigenous to South Africa, are stated to be eaten by both the natives and animals of the South-West Protectorate of the Union of South Africa. No analysis of the beans appears to be on record, and the following results of the examination of a sample recently received at the Imperial Institute from South Africa are therefore of interest.

The beans were dark reddish-brown, roughly spherical in shape, and about $\frac{1}{2}$ to $\frac{3}{4}$ in. in diameter. They had a woody shell, the inner surface of which was covered with a thin layer of opaque, tough, white, horny material. The kernel was fairly hard, cream-coloured, and oily, and had a pleasant nutty flavour with a faint bitter taste. The beans were composed of 49 per cent. of shell and 51 per cent. of kernel. The average weight of a bean was 2 grms. and that of a kernel 1 gram.

The results of the examination of the beans are given below :

	Moisture.	Crude Proteins.	Fat (extd. with sodium-dried ether).	Carbo-hydrates, etc. (by difference).	Crude Fibre.	Ash.	Nutrient Ratio.	Food Units
Husks alone	(per cent.) 8.5	2.5	0.2	67.2	19.8	1.8	1 : 27.1	74
Kernels alone	4.0	32.8	41.6	17.2	1.3	3.1	1 : 3.4	203
Whole beans	6.2	18.0	21.3	41.6	10.4	2.5	1 : 5.0	140

The beans are free from alkaloids and cyanogenetic glucosides.

A quantity of oil was extracted from the kernels with petroleum spirit, and was a golden yellow, limpid liquid with a nutty odour and an agreeable taste. On analysis the following figures were obtained :

Specific gravity at 15/15° C. ...	0.9211
Acid value ...	0.6
Saponification value ...	190.0
Iodine value (Hübl, 17 hours) ...	95.6
Volatile acids (soluble*) ...	0.3
Volatile acids (insoluble*) ...	0.1
Unsaponifiable matter, per cent. ...	0.8
Refractive index, (n) _D ²⁰ ...	1.464
"Titre" of fatty acids °C. ...	30.6

The residual cake left after the expression of the oil would have the following composition (when calculated on the basis of a 7 per cent. oil content): Moisture, 6.4; crude proteins, 52.2; fat, 7.0; carbohydrates, etc. (by difference), 27.4; crude fibre, 2.1; and ash, 4.9 per cent. Nutrient ratio, 1 : 0.83; food units, 175.

The above examination shows the kernels to be rich in crude proteins and oil. The oil is of good appearance, and has constants very similar to those of cottonseed

* Number of c.c. $\frac{N}{10}$ potassium hydroxide solution required to neutralise the acids from 5 grms. of oil.

oil, and should be of value as an edible oil. The cake left after the expression of the oil contains a very high percentage of protein and a low amount of crude fibre. Its value would probably be well above that of decorticated cottonseed cake, but it would be advisable to carry out feeding trials with it before making a definite recommendation.

In India the seeds of *Bauhinia esculenta*, and also those of other species of *Bauhinia*, are employed as food, whilst those of *B. VahlII* are stated to have medicinal properties.

The author desires to acknowledge his indebtedness to Mr. P. F. C. Sowter, B.Sc., A.I.C., for his assistance in carrying out some of the work.

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NOTES.

The Editor desires to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

A ROUTINE TEST FOR THE PRESENCE OF SULPHITES.

THE following method has been found to be a useful routine one for the detection of sulphites added as a preservative or bleaching agent to foodstuffs, confectionery, and other goods.

It is a modification of the combined methods of Schmidt (*Arbeiten aus dem Kaiserlichen Gesundheitsamte*, 21, 226) and of Winton and Bailey (*J. Amer. Chem. Soc.*, 1907, 29, 1499), and in practice has been found to be speedy, sensitive, and efficient, without the disadvantages of the better-known method of reduction by means of zinc to hydrogen sulphide (*U.S. Dept. Agr. Bul.*, 107, A.O.A.C.).

Ten grms. of the material, such as dried fruit or minced meat or fish, are incorporated with 10 to 20 c.c. of water, by means of a pestle and mortar, and transferred to a small conical flask of about 50 c.c. capacity. In the case of fruit-pulp, glucose-syrup, or fruit juice, 10 c.c. may be diluted, when necessary, with 10 to 20 c.c. of water in the flask.

Ten c.c. of dilute sulphuric acid of about 2N-strength and two or three small fragments of marble chips are now introduced into the flask, and the mouth immediately covered with a piece of starch paper (impregnated with a 1 per cent. starch solution), which should be screwed round the neck of the flask, and held in place with a rubber ring. The reason for the addition of the marble is to set up a gentle current of carbon dioxide to sweep out the oxygen and the liberated sulphur dioxide. The top of the paper is moistened with 1 drop of a 1 per cent. solution of iodine.

In the presence of any appreciable quantity of sulphites the blue stain on the starch paper will be immediately discharged by the sulphur dioxide. If traces only be present, it may take a few minutes. The action takes place in the cold; it may be hastened by leaving the flask in a warm place.

If the drop of iodine solution used be of the magnitude of 0.1 c.c. it is obvious that the limit of sensitiveness of the test is the quantity of sulphur dioxide necessary

to reduce the iodine and discharge the blue colour—namely, 0·00025 grm.; and this is the limit usually found when using known amounts of sulphites, showing that practically the whole of the liberated gas is driven out of the flask. This amount, if 10 grms. of the material be taken, would represent 0·0025 per cent. of sulphur dioxide, 0·175 grain per lb., or 1·75 grains per gallon respectively.

By using a weaker solution of iodine the test could be made more sensitive, but for a routine qualitative test the strength suggested makes it sufficiently delicate for the amounts usually met with.

Traces of hydrogen sulphide do not seriously interfere with this method, but in practice 1 c.c. of a 5 per cent. solution of copper sulphate is added to the other ingredients when testing meat or fish, and this will retain as much hydrogen sulphide as is likely to be present.

ALBERT E. PARKES.



NOTES FROM REPORTS OF PUBLIC ANALYSTS.

The Editor would be glad to receive the Annual or other Reports of Public Analysts containing matter of interest to the Society. Notes made from such Reports would be submitted to the Publication Committee.

BOROUGH ANALYST'S REPORT TO THE BOROUGH OF SALFORD FOR THE YEAR 1920.

VISITATION OF FARMS.—It is considered that the primary object of the department is to procure a pure milk supply, and that legal proceedings should only be taken in cases of apparent fraud, or where persuasion fails to effect an improvement. Hence, during the year 1920, the proportion of milk dealers personally interviewed has been much larger, whilst in cases where farm samples have been unsatisfactory the farms have been visited with the object of finding the cause, and, if possible, removing it.

"APPEALS TO THE COW."—In the case of twelve samples of milk deficient in fat by amounts varying from 6 to 15 per cent. "appeal to cow" samples were taken by the inspector. These were found to be deficient in fat, although the solids-not-fat figures were up to the average. The farm was therefore visited by the Veterinary Surgeon and the Borough Analyst. There were thirty cows, all in fair condition, yielding about twenty-six gallons of milk. The feed consisted largely of brewers' grains and turnips, with a proportion of maize meal and oats. It was considered that the albuminoid ratio was too low, and suggestions for improvement were made to the farmer. As a result, the milk supply has been permanently improved. After five weeks, four "appeal to cow" samples were again taken, and were found to contain 3·6, 3·0, 3·4, and 3·4 per cent. of fat respectively.

DIRT IN MILK.—The following method of estimation is used: The milk (500 c.c.) is allowed to stand for some hours in a cylindrical vessel, the bottom of which is drawn out and fitted with a tube graduated in 0·01 c.c. The volume of sediment is read directly and multiplied by two to obtain the parts per 100,000 of volume of sediment in the milk. Of the 613 samples examined, 396 gave no measurable sediment, 98 gave a sediment of 0·5 per 100,000, 62 gave 0·1, 3 gave 1·5, 29 gave 2·0, 6 gave 3·0, 1 gave 4·0, 6 gave 5·0, 3 gave 6·0, 2 gave 7·0, 3 gave 9·0, 1 gave 10·0, 1 gave 12·0, 1 gave 15·0, and 1 gave 20·0. As a provisional standard, samples giving a sediment of more than 5 volumes per 100,000 are being regarded as adulterated, although obviously this is too lenient to be accepted as a permanent standard.

SAMPLES UNDER THE RAG FLOCK ACT, 1911.—Three samples were found to contain 70, 50, and 100 parts respectively of soluble chlorine per 100,000, and there-

fore did not conform with the requirements for the standard of cleanliness fixed by the Ministry of Health in the Rag Flock Regulations, 1912 (viz., a maximum of 30 parts per 100,000). In each case a circular letter was sent by the Medical Officer of Health to the vendor, intimating that in future proceedings would be taken where the flock fell below the legal standard.

G. D. ELDON.



ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Discoloration of Sweet Potatoes in Tins. E. F. Kohman. (*J. Ind. Eng. Chem.*, 1921, 13, 634, 635.)—The black coloration sometimes observed in tinned sweet potatoes is due to combination of a tannin-like constituent with iron derived from the container; since the iron must be in the ferric state in order to give the coloration, the access of air is necessary for the formation of the latter, and the necessity of tight seams is emphasised.

W. P. S.

Eggs and Egg Products. (*Report of Connecticut Agric. Exper. Stat. Bull.*, 227, 1921, 223-224.)—*Composition of Egg-Shells*: Air-dried egg-shells (without the inner membranous lining) contained 0.21 to 0.64 per cent. of moisture (15 samples), 49.40 to 53.28 per cent. of lime as CaO (26 samples), and 1.04 to 2.48 of magnesium oxide (7 samples), and lost on ignition 45.41 to 46.39 per cent. (15 samples). *Egg-Powder*: A sample stated to consist of sprayed egg yolk, albumin, salt, powdered skimmed milk, starch, gelatin, and sodium bicarbonate, had the following composition: Moisture, 6.83; ash, 9.47; protein ($N \times 6.25$), 20.48; starch, 26.40; fat, 13.85; and lecithin phosphoric anhydride, 0.46 per cent. No artificial colour was present. *Egg "Noodles"*: Eight samples gave the following results on analysis: Ash, 0.68 to 1.66; protein, 12.94 to 16.56; and lecithin phosphoric anhydride, 0.028 to 0.066 per cent. They were free from artificial colour. The lecithin phosphoric acid (P_2O_5) content was taken as the index of egg material present. Accepting Juckenack's standard (*Conn. Exp. Stat. Record*, 1904, p. 138), 0.0225 per cent. of lecithin phosphoric acid may be found in "noodles" prepared without eggs, and this amount is more than doubled by the addition of egg or egg yolk in the proportion of one to 1 lb. of flour. Figures less than 0.035 or 0.040 per cent. do not indicate appreciable amounts of eggs. *Dehydrated Eggs*: Two samples gave the following results: Moisture, 6.25 and 6.62; ash, 3.42 and 3.50; proteins, 40.44 and 43.00; fat, 45.12 and 42.29; and lecithin P_2O_5 , 1.37 and 1.33 per cent.

Adulteration of Egg-Powder with Cereal. (*U.S. Dept. Agric. Bureau of Chemistry. Service and Regulatory Announcements*, June, 1921. Suppl. 113, 96-97.)—An article sold as "whole egg-powder made strictly from fresh eggs" was found to consist essentially of maize starch, egg albumin, and a small amount of calcium and

aluminium phosphates, and to be artificially coloured with tartrazine. It was condemned in the District Court of the United States as adulterated, and also as misbranded, notwithstanding the fact that the following statement was inconspicuously printed on the label: "Artificially coloured, The contents of this packet contains powdered yolk of fresh eggs, albumin, corn starch, and phosphate."

Ice Cream. (*Connecticut Agric. Exper. Stat. Bull.*, 227, 1921, 236-249.)—According to the Public Acts of 1919, ice cream manufactured in the State of Connecticut must contain not less than 8 per cent. of milk fat in (plain) ice cream, and not less than 6 per cent. in fruit and nut ice cream. It must be free from boric acid, salicylic acid, formaldehyde, saccharin, salts of copper, iron oxide, ochres, and injurious colours or flavours. Harmless permitted colours and flavours must be declared, and the use of harmless vegetable gums and gelatin is allowed. Ice cream containing less fat than the standard amounts may be sold provided the true fat content is declared. Of 400 samples examined in 1920, forty-seven contained less than the standard amounts of fat, but the average proportion was 8.8 per cent.

Estimation of Caffeine in Tea and Coffee. R. E. Andrew and E. M. Bailey. (*Connecticut Agric. Exper. Stat. Bull.*, 227, 1921, 256-258.)—The following simple method, based on the methods of Power and Chestnut (*J. Amer. Chem. Soc.*, 41, 1300), of Stahlschmidt (*J. Amer. Off. Agric. Chem.*, 2, 3, 332), and of Deker (*Chem. Zentrbl.*, 1903, 1, 1, 62), is submitted for collaborative study with a view to its adoption as an optional official process: Five grms. of the tea or coffee, previously ground to pass through a $\frac{1}{32}$ in. sieve, are boiled with 10 grms. of heavy magnesium oxide in a 500 c.c. flask for two hours over a low flame, a narrow glass tube 30 ins. long being used as condenser. The contents of the flask are then cooled, made up to 500 c.c. and filtered, and 300 c.c. of the filtrate mixed with 10 c.c. of 10 per cent. sulphuric acid, and evaporated to about 100 c.c. in an Erlenmeyer flask. The liquid is filtered into a separating funnel, the flask being rinsed out with small portions of 1 per cent. sulphuric acid, and the whole then shaken with six successive portions of chloroform (25, 20, 15, 10, and 10 c.c.). The united extracts are treated with 5 c.c. of a 1 per cent. solution of potassium hydroxide, and the chloroform layer is subsequently drawn off, whilst the alkaline layer is washed with two portions (10 c.c. each) of chloroform, and the washings added to the main extract. The chloroform is distilled in a weighed flask, and the residue dried at 100° C. until constant in weight. If desired, the nitrogen in the residue may be estimated by Kjeldahl's method, and the caffeine calculated from the nitrogen multiplied by the factor 3.464. The quoted results obtained with various kinds of tea and with coffee by either method agree closely with those obtained by the more tedious methods of Stahlschmidt and of Power and Chestnut.

New Method for the Volumetric Estimation of Reducing Sugars. A. Jonescu and V. Varcolici. (*Ann. Chim. anal.*, 1921, 3, 229-234.)—The reagent employed contains 46 grms. of potassium ferricyanide and 46 grms. of potassium hydroxide per litre; it is standardised by titration with 0.5 per cent. dextrose

solution, 10 c.c. of the reagent being mixed with 20 c.c. of water and heated to boiling, and the sugar solution added, drop by drop, from a burette until the yellow colour of the ferricyanide has disappeared completely. Ten c.c. of the reagent should be reduced by 0.05 gm. of dextrose. The method may be applied to the estimation of lactose in milk, after this has been treated with acetic and picric acids, filtered, and the filtrate neutralised. When the concentration of the sugar solution is much less than 0.5 per cent., it is advisable to boil a portion of the sugar solution with the reagent, and to titrate the ferrocyanide with permanganate in acid solution. One litre of *N*-permanganate solution is equivalent to 368.34 grms. of potassium ferrocyanide, 329 grms. of potassium ferricyanide, or 30 grms. of dextrose.

W. P. S.

Estimation of Maltose and Lactose in the Presence of other Reducing Sugars by Barfoed's Reagent. L. Le Grand. (*Ann. Chim. anal.*, 1921, 3, 240-244.)—Barfoed's reagent is prepared by dissolving 20 grms. of neutral copper acetate in 300 c.c. of water, adding 7.5 c.c. of 38 per cent. acetic acid, and filtering the mixture. It is reduced by dextrose, lævulose, and galactose, but not by maltose and lactose. To estimate the two latter in the presence of other reducing sugars, a mixture of 5 c.c. of the sugar solution (containing not more than 0.1 gm. of total sugars) and 15 c.c. of the reagent is boiled for three minutes, filtered, the cuprous oxide is dissolved in ferric sulphate solution, and the quantity of ferrous sulphate formed is found by titration with standardised permanganate solution. Each c.c. of the latter solution should be equivalent to about 0.006 gm. of copper. Reference to a table gives the quantity of monoses corresponding with the amount of cuprous oxide formed. The total quantity of reducing sugars is estimated in another portion of the original sugar solution by means of Fehling's solution; the difference between the two estimations gives the amount of maltose and lactose present.

W. P. S.

Detection and Estimation of Lævulinic Acid in Foods. L. Grünhut. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1921, 41, 261-280.)—A very sensitive reaction for lævulinic acid (β -acetylpropionic acid) depends on the red coloration obtained when a dilute solution of the acid is treated with sodium nitro-prusside; the coloration is stable in alkaline and in acetic acid solution. Lævulinic acid is generally accompanied in foods by formic acid, and sometimes by acetic acid and lactic acid. To estimate these acids, the sample is acidified with phosphoric acid and extracted with ether; the extraction-flask should contain a quantity of sodium hydroxide solution to prevent volatilisation of the formic acid with the ether vapour. The ethereal extract is then shaken with dilute sodium hydroxide solution, the aqueous portion separated, evaporated to dryness, and the residue dissolved in a definite volume of water. If formic and lævulinic acids alone are present, the formic acid is estimated in a portion of the solution by the mercuric chloride method; another portion (50 c.c.) of the solution is then heated under a reflux apparatus for two hours with 20 c.c. of dilute sulphuric acid and an excess of potassium dichromate solution; the formic acid is oxidised to carbon dioxide and water, whilst the lævulinic acid is oxidised to carbon dioxide, water, and acetic acid, according to the equation:

$C_5H_8O_3 + 7O = C_2H_4O_2 + 3CO_2 + 2H_2O$. The mixture is then distilled, and the acetic acid titrated in the distillate; the amount of dichromate reduced corresponds with the sum of the quantities of formic and acetic acids. When the sample itself contains acetic acid, the amount found in the distillate will be correspondingly more than is indicated by the amount of dichromate reduced. If all four acids (formic, acetic, lævulinic, and lactic) are present, it is advisable to submit the solution to a simple distillation. The distillate will contain almost the whole of the acetic acid and most of the formic acid, but practically no lactic acid, whilst the residual solution will contain all the lævulinic acid, the lactic acid, and a portion of the formic acid. The two solutions are then analysed separately as described, oxidation with dichromate giving the amount of acetic acid corresponding with the lactic acid, as shown by the equation: $C_3H_6O_3 + O_2 = C_2H_4O_2 + CO_2 + H_2O$. To ensure complete oxidation of the lactic acid, the heating must be continued for a further two hours after 30 c.c. of concentrated sulphuric acid have been added to the mixture already oxidised as described.

W. P. S.

Composition of Hollyhock Seed and Oil. R. S. Hiltner and L. Feldstein. (*J. Ind. Eng. Chem.*, 1921, 13, 635.)—Mature seeds from the pods of different varieties of hollyhock had the following composition: Water, 4.4; ash, 6.9; oil (ether extract), 11.9; crude protein, 21.2; crude fibre, 25.6; and starch, 9.1 per cent. The oil had a greenish-yellow colour; its physical and chemical characters were: Specific gravity at 15.6° C., 0.9275; n_D^{25} , 1.4722; and iodine value, 119.0. With Halphen's and Bechi's tests the oil gave positive reactions, thus resembling cottonseed oil; the coloration with Halphen's reagent was not obtained when the oil had been heated previously for ten minutes at 250° C.

W. P. S.

Vanilla Paste. (*Connecticut Agric. Exper. Stat. Bull.*, 227, 1921, 258.)—Vanilla extract of standard quality should contain in 100 c.c. the soluble matter from not less than 10 grms. of vanilla bean (*Standards of Purity for Food Products*, U.S.D.A. Circ. 136 [1919]). The amount of vanillin obtained from 10 grms. of vanilla bean will depend upon the quality of the bean, and may vary from 0.07 to 0.24 gm. (*Conn. Exp. Stat. Report*, 1901, p. 150). A sample of vanilla paste recently examined consisted of gum, glycerin, and sugar, with 0.047 gm. of vanillin. The contents of the tube were stated to be equal to 1 pint of liquid extract of vanilla, and therefore should have contained at least 0.33 gm. of vanillin in the total quantity of 36.7 grms.

Derivatives of Sulphur in Commercial Salvarsan. H. King. (*J. Chem. Soc.*, 1921, 119, 1107-1120.)—The difficulty experienced by manufacturers in preparing salvarsan of uniform toxicity is due to the substance being amorphous or doubtfully crystalline, and so not readily purified, and secondly to the use of sodium hyposulphite in the reduction process. Solutions of commercial salvarsan in methyl alcohol yield an insoluble deposit, which Fargher and Pyman (*J. Chem. Soc.*, 1920, 117, 373) regarded as the monohydrochloride of the monosulphamic acid of 3:3'-diamino-4:4'-dihydroxyarsenobenzene. An abnormal commercial sample recently examined yielded, when dissolved in methyl alcohol, 24 per cent. of a deposit which

had a similar elementary composition to that of Fargher and Pyman's preparation, but examination of several samples of the insoluble constituent showed that, in general, it consisted of a mixture of 3 : 3' - diamino - 4 : 4' - dihydroxy - 5 - sulphino-arsenobenzene monohydrochloride and "salvarsan sulphate" and "hydrochloride," the two latter probably being present chiefly in the form of the mixed salts. There was no evidence of the presence of a monosulphamic acid. The reduction of the basic property of the salvarsan molecule by the introduction of a sulphinic or sulphonic group has a distherapeutic effect, the influence of the sulphonic group being somewhat greater than that of the sulphinic group. The greater relative efficacy of pure sulphur-free salvarsan, prepared by means of hypophosphorous acid, compared with that of monosulphino-arsenobenzene, is shown by the results (described in detail) obtained by intravenous injection of the preparations into mice.

Detection of Neosalvarsan in a Complex Mixture. M. A. Morel. (*Ann. Chim. anal.*, 1921, 3, 215-216.)—In the case of a mixture of iodides of mercury and potassium, sodium bicarbonate, arrhenal, and vegetable substance of brown colour, supposed to contain 0.6 per cent. of neosalvarsan, it was impossible to detect novo-arsenobenzene owing to the fact that insoluble brown substances masked the colour reactions in acid and alkaline solutions. The mixture was therefore examined (a) alone, (b) after adding a quantity of novo-arsenobenzene equivalent to the amount supposed to be present. Three grms. were acidified with dilute hydrochloric acid (1 to 10) and treated with dimethylaminobenzaldehyde in hydrochloric acid. After twenty-four hours the precipitate was centrifuged off, washed free from acid and arrhenal, and, after destruction of organic matter, tested for arsenic in Marsh's apparatus. Arsenic was thus detected in the mixture to which novo-arsenobenzene had been added (b), but not in the mixture itself (a). A test for formaldehyde was also made in the distillates obtained after treatment with dilute sulphuric acid; formaldehyde was detected in (b), but not in (a). (Cf. *ANALYST*, 1921, 144.)

R. G. P.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Substance yielding Hydrogen Sulphide contained in the Seeds of Certain Papilionaceæ. M. Mirande. (*Comptes rend.*, 1921, 173, 252-253.)—Further investigation supports the view that the source of the hydrogen sulphide yielded by the auto-fermentation of *Lathyrus* and other members of the *Papilionaceæ* family (*ANALYST*, 1921, 291) is a substance of protein character; only part of the total sulphur present is emitted as hydrogen sulphide.

T. H. P.

Investigation of the Products of the Enzymic Hydrolysis of Inulin by the Biological Method for the Detection of Dextrose. E. Bourquelot and M. Bridel. (*J. Pharm. Chim.*, 1921, VII., 24, 81-89.)—It has been found previously that, when dextrose is dissolved, either alone or together with other sugars, in 70 per cent. aqueous methyl alcohol, 82.6 per cent. of it is transformed into β -methylglucoside by the action of emulsin (*ANALYST*, 1920, 45, 175, 413). Application of this

method of investigation to the product resulting from the hydrolysis of inulin by the inulase of *Aspergillus niger* shows that this product consists solely of lævulose.

T. H. P.

Occurrence of Manganese throughout the Vegetable Kingdom. G. Bertrand and M. Rosenblatt. (*Comptes rend.*, 1921, 173, 333-336.)—Maumené (*Comptes rend.*, 1884, 98, 1416) gave a list of plants which, contrary to the general rule, he stated to be free from manganese. These included oranges, lemons, onions, and certain cruciferous plants. The authors have investigated these supposed exceptions, and in all cases found manganese to be present in appreciable proportions. For example, in the case of a lemon the rind contained 0.17; the white pith, 0.07; the pulp (without pips), 0.03; and the pips, 0.24 mgrm. of manganese per 100 grms.

T. H. P.

Relative Digestibility of Various Preparations of Proteins from Beans. H. C. Waterman and D. B. Jones. (*J. Biol. Chem.*, 1921, 47, 285-295.)—Experiments *in vitro* and *in vivo* on the proteins of Chinese and Georgia velvet beans (*Stizolobium niveum* and *S. deeringianum*) show that the dialysed proteins are not sufficiently digestible for animal nutrition, but that when the dialysed protein is coagulated by boiling the increased digestibility is sufficient for animal growth. The raw and cooked beans contain dihydroxyphenylalanine, which also is a limiting factor. The digestibility of the proteins *in vitro* in terms of amino-nitrogen is 30 per cent., which is increased to 57 per cent. (determined by digestion with pepsin and trypsin) on coagulation. Parallel experiments *in vivo* lead to the conclusion that whilst there is no definite quantitative relationship between digestibility *in vitro* and *in vivo*, there is a general agreement and rough proportionality, and variations are in the same direction. To account for the indigestibility of certain proteins, it is thought that the amino-acid residues are united in a different order from that in others which are readily assimilated by the animal and which are acted upon by the proteoclastic enzymes.

H. E. C.

Viscous Lactic Streptococcus in Milk. H. Violle. (*Ann. Inst. Pasteur*, 1921, 35, 218-229.)—An objectionable form of lactic streptococcus is frequently present in milk and milk products. When grown on nutrient gelatin it forms large aggregates of crystalline appearance, whilst in saccharine media it produces lactic acid at the expense of most of the sugar, and yields a glairy substance. It is not pathogenic, and may be distinguished from the pathogenic streptococci of man and of the cow by its biological characteristics and the appearance of its cultures. It also differs in certain particulars from ordinary lactic streptococci, and ought, therefore, to be classified with the paralactic or pseudolactic bacteria.

Toxicity of Various Fractions and Combinations of Fractions of Coal Tar Creosote to Wood-destroying Fungi. H. Schmitz and S. M. Zeller. (*J. Ind. Eng. Chem.*, 1921, 13, 621-623.)—The results obtained in the investigation recorded show that there is no visible cessation of growth of either *Lenzites sapinaria* or *Poly-*

porus lucidus when the concentration of the coal tar creosote is below 1 per cent. In the majority of cases, the toxic point (the minimum percentage of creosote which will inhibit completely the growth of the organisms) lies between 2 and 4 per cent. The fact that these results are at variance with those recorded by other observers is due to the methods used and to the disturbing influence of many factors. It is evident that *Lenzites sapinaria* is more resistant to greater concentrations of creosote than is *Polyporus lucidus*. As would be expected, the toxic point varies with the nature of the wood. The most toxic fractions are those which distil between 235° and 270° C. and between 270° and 315° C.; there is no great difference in the toxicity of any of the combinations of the various fractions except when the residue above 355° C. is omitted. This residue is toxic only in very high concentration. The method employed for determining the toxic value consisted, briefly, in impregnating dry sawdust with an alcoholic solution of the creosote, and exposing the impregnated sawdust to the atmosphere for twenty-four hours to allow all the alcohol to evaporate; 3 grms. of the treated sawdust were then saturated with water in a flask, sterilised in an autoclave, cooled, and inoculated with the cultures. The computed percentages were calculated on the dry sawdust, without taking into account the water present during the growth of the organisms; in commercial practice, the amount of impregnation is calculated as lbs. of creosote per cb. ft. of wood. Impregnated timbers, such as railway sleepers, may be placed under conditions, as regards moisture, very favourable to the growth of fungi, and the tests were intended to imitate these natural conditions.

W. P. S.

Toxicity of Wood Preservatives. C. J. Humphrey, R. M. Flemming, and E. Bateman. (*J. Ind. Eng. Chem.*, 1921, 13, 618-621.)—The toxicity of a number of wood preservatives was tested by means of a method described previously (*ANALYST*, 1915, 40, 447), the fungus used, *Fomes annosus*, being incubated at 26° C. on agar medium containing varying quantities of the preservatives. With beechwood creosote, the toxicity of the fractions increased with the boiling-point, that of the fraction boiling at 225° C. having, at a concentration of 0.05 per cent., five times the killing power of the fraction boiling at 180° C. The toxicity of various commercial preservatives corresponded with their cresylic acid content. The most toxic substance examined was a preparation containing about 24 per cent. of dinitrophenol; this, in a concentration of less than 0.01 per cent., inhibited the growth of the organism.

W. P. S.

Yeast Test as a Measure of Vitamin-B. W. H. Eddy, H. L. Heft, H. C. Stevenson, and R. Johnson. (*J. Biol. Chem.*, 1921, 47, 249-275.)—A critical study of yeast stimulation as a method of testing for water-soluble vitamin-B has been made, the technique of Funk and Dubin (*J. Biol. Chem.*, 1920, 44, 487) being used with various extracts; it is found that, owing to lack of a basal medium providing an optimum of all factors except vitamin-B, the test is of little value as an estimation of vitamin content, as compared with rat-feeding experiments, though there is an approximate agreement if the extracts are dilute. When the solution is

very dilute, a destructive effect, due to the alkali, begins to appear, but the results are very contradictory. (*Cf. ANALYST*, 1921, 146, 246.) H. E. C.

Fat-Soluble Vitamin and Yellow Pigmentation in Animal Fats. H. Steenbock, M. T. Sell, and M. V. Buell. (*J. Biol. Chem.*, 1921, 47, 89-109.)—It was considered that fat-soluble vitamin might be identical with yellow pigments of the carotinoid type, but experiment shows this is not so (*cf. ANALYST*, 1921, 246). In cod-liver oil there is much fat-soluble vitamin, with very small amounts of yellow, pigments. In butter there is a seasonal variation in fat-soluble vitamin content, which does not run parallel with its yellow pigment; but, in general, owing to the content of the cow-feeds, highly pigmented butters are rich in the vitamin. Pale butters are looked upon with suspicion, and may contain as little vitamin as oleo-margarine. In the case of beef-fats, the most pigmented samples are generally the richest in the vitamin. The fact that fat-soluble vitamin is resistant to saponification by severe methods shows that it is not a fat or an ester. H. E. C.

Fat-Soluble Vitamin Content of Peas in Relation to their Pigmentation. H. Steenbock, M. T. Sell, and P. W. Boutwell. (*J. Biol. Chem.*, 1921, 47, 303-308.)—Following the author's theory that fat-soluble vitamin is related to certain yellow-plant pigments, it is shown that in the case of ripe peas, out of six samples investigated the green peas which contained a quantity of yellow pigment were far richer in content of fat-soluble vitamin than yellow peas which contained much less yellow pigment. H. E. C.

Anti-scorbutic Principle of Potato Juice. Bezssonoff. (*Comptes rend.*, 1921, 175, 417-419.)—Raw uncrushed potato has a strong anti-scorbutic power, but the expressed juice has only a weak action, which is only slightly intensified by the addition of the residual *marc*. This instability of the anti-scorbutic principle appears to be due to enzymic action, and, in particular, to an oxydase (Bertrand's laccase). By expressing the juice from the potato in the presence of citric or tartaric acid the action of the laccase is inhibited, and a product which does not turn brown on exposure to air and has a strong anti-scorbutic power is obtained. The juice expressed from new potatoes has a much more pronounced anti-scorbutic acid than that derived from old potatoes.

Effect of Heat and Oxidation upon the Anti-scorbutic Vitamin. R. A. Dutcher, H. M. Harshaw, and J. S. Hall. (*J. Biol. Chem.*, 1921, 47, 483-488.)—Experiments previously made indicated that rhubarb juice and orange juice could be boiled for fifteen minutes without losing their anti-scorbutic power. Analogous results were obtained by Delf (*Biochem. J.*, 1920, 14, 211) with juices extracted from fresh cabbages, Swedish turnips, and oranges, and it was suggested that the stability of the anti-scorbutic vitamin at temperatures above 100° C. might be due to the absence of air. Further experiments have shown that the anti-scorbutic vitamin in fresh orange juice is not destroyed by pasteurisation for thirty minutes at 63° C. in closed vessels, or by boiling for thirty minutes beneath a reflux condenser.

Hydrogen peroxide has some destructive influence on the vitamin when added to the juice at the ordinary temperature, and this action is intensified by heating the mixture of orange juice and hydrogen peroxide at 63° C. and 100° C. Hence, the conclusion is drawn that the anti-scorbutic properties of orange juice are susceptible to oxidation, but, in the absence of oxidising agents, are stable at temperatures up to the boiling-point of the juice.

Estimation of the Diffusible Calcium of Blood Serum. L. v. Meysenbug, A. M. Pappenheimer, T. F. Zucker, and M. F. Murray. (*J. Biol. Chem.*, 1921, 47, 529-537.)—From 30 to 40 per cent. of the calcium in ox serum is in a non-filterable colloidal form and apparently in combination with proteins (Cushny, *J. Physiol.*, 1919-20, 53, 391). If blood serum be dialysed against a calcium-free Ringer's buffer solution (NaCl, 36.0; NaHCO₃, 10.08; KCl, 1.68 grms., and water to 2,000 c.c.), there appears to be progressive dissociation of the colloidal calcium compound, until, after seven days, about 90 per cent. of the total calcium will have passed into the dialysate. When, however, calcium is added to the dialysing fluid equilibrium is obtained within twenty-four hours. For this purpose an equal volume of standard calcium chloride solution containing approximately the same amount of calcium as the serum (10.5 to 11 mgrms. per 100 c.c.) was mixed with the dialysing fluid, which, like the serum, had previously been saturated with a mixture of carbon dioxide and air of known tension. The added calcium was deducted from the amount estimated in the dialysate, and the result deducted from the total amount in the original serum. By this method the diffusible calcium of the serum of normal men and dogs was found to vary from 60 to 70 per cent. of the total serum calcium.

Gasometric Estimation of Urea in Urine. R. L. Stehle. (*J. Biol. Chem.*, 1921, 47, 13-17.)—The following is a rapid and accurate method for estimating urea in urine: Ammonium salts are removed by treatment of 25 c.c. of the diluted urine (1:10) with 4 grms. of permutit for four minutes; 1 c.c. of the filtered ammonia-free urine is transferred to Van Slyke's carbon dioxide apparatus, rinsed in with 1 c.c. of water, followed by 1 c.c. of sodium hypobromite solution. A correction is made for the gases contained in the urine, rinsing water, and hypobromite solution; the solubility of air in diluted urine is the same as in water, and the hypobromite solution contains 0.006 c.c. at 15° C. to 20° C. and 0.005 c.c. at 21° C. to 25° C. Nitrogen is liberated quantitatively from the urea in about half a minute, but not appreciably from other nitrogenous urinary constituents. H. E. C.

Zinc-Potassium Ferrocyanide as a Reagent in Urine Analysis. Thiéry. (*J. Pharm. Chim.*, 1921, 23, 494-503.)—The xanthine bases and uric acid are estimated together in 200 c.c. of urine and expressed as uric acid. In another 200 c.c. of the urine the xanthine bases are precipitated with 20 c.c. of potassium ferrocyanide solution (150 grms. anhydrous salt per litre), followed by 20 c.c. of zinc acetate solution (112 grms. anhydrous salt per litre acidified with 5 to 10 c.c. of glacial acetic acid), the precipitate is separated, and 120 c.c. of filtrate (= 100 c.c. of urine) are used for the estimation of uric acid by Haycraft and Denigès' method

(*cf.* ANALYST, 1896, 21, 212). The difference between the amount of uric acid found and the amount of (apparent) uric acid found in the original untreated sample is calculated as xanthine. The precipitate given by potassium ferrocyanide and zinc acetate contains all the creatinine and xanthine bases, and most of the alloxan, alloxantin, and allantoin, whilst all the uric acid passes into the filtrate.

R. G. P.

Dinitrosalicylic Acid as a Reagent for the Estimation of Sugar in Urine.

J. B. Sumner. (*J. Biol. Chem.*, 1921, 47, 5-9).—The method is based on the fact that 3, 5-dinitrosalicylic acid is reduced by glucose to a highly-coloured nitroamino compound, but it is not reduced by other urinary constituents, except when glucose is present. A correction may be applied for the reduction which even normal urine produces after the destruction of its sugar by boiling with caustic alkali. Dinitrosalicylate solution is prepared containing 2 per cent. of the acid and 2 per cent. of sodium carbonate, and also standard glucose solutions containing 1 and 0.5 mgrm. per c.c. preserved with toluene. For the estimation 1 c.c. of the dinitrosalicylate solution and 2 c.c. of 1.5 per cent. sodium hydroxide solution are added to 1 c.c. of the clear urine (which, if containing more than 0.4 per cent. of sugar, must be diluted), and the test tube is plugged with wool and heated in boiling water for five minutes. The cool mixture is diluted in volume to match the colour of a standard similarly prepared from 1 c.c. of the stronger (1 mgrm.) glucose solution. To correct for the reducing action of uric acid and polyphenols in normal urine, heat 1 c.c. of the urine in boiling water for fifteen minutes with 1 c.c. of 3 per cent. sodium hydroxide solution, cool, add 1 c.c. of glucose solution (containing either 1 mgrm. or 0.5 mgrm., according to whether the total reduction was equivalent to more or less than 1 mgrm. per c.c.) and 1 c.c. of dinitrosalicylate solution, and proceed as before. The amount of glucose indicated, less the amount added, gives the reducing value due to the urine as distinct from its sugar. Details are given for preparing the 3, 5-dinitrosalicylic acid.

H. E. C.

Estimation of Veronal. **L. Van Itallie and A. J. Steenhauer.** (*Pharm.*

Weekblad, 1921, 58, 1062-1068.)—For the separation of veronal from urine, ethyl acetate is preferable to ether, the solubility of veronal in this solvent being 1 : 8.9. A preliminary treatment of the urine with lead acetate or basic lead acetate effects precipitation of some of its constituents and prevents emulsification during the solvent extraction. The use of charcoal for purifying the extracted veronal leads to low yields on account of adsorption; treatment with potassium permanganate gives quantitative results. The method of estimation is as follows: 100 c.c. of urine are mixed with 10 c.c. of lead acetate, and filtered, and 100 c.c. of the filtrate are evaporated to 25 c.c. and acidified with acetic acid. The solution is shaken with twice its volume of ethyl acetate, and filtered, and the residue, after distillation of the solvent, is dissolved in 10 c.c. of boiling water, 5 c.c. of dilute sulphuric acid are added, and the heated mixture is treated with $\frac{N}{10}$ potassium permanganate until the supernatant liquid above the precipitate becomes colourless. The manganese oxide which separates is decomposed with a few drops of hydrogen peroxide, the solution is again

shaken with ethyl acetate, filtered, and evaporated, and the residue dried at 100° C. For estimating veronal in human organs, these are extracted with alcohol in presence of acetic acid. After filtration, and evaporation to about 25 c.c., absolute alcohol is added until no more precipitate forms, and the solution is again filtered and evaporated. The residue is boiled with water, filtered while hot, and shaken twice with ethyl acetate. The further treatment is carried out as described above. W. J. W.

AGRICULTURAL ANALYSIS.

Relation of Hardness to Protein Content of Wheat. H. F. Roberts. (*J. Agric. Research*, 1921, **21**, 507-522.)—There is some indirect evidence in support of the commonly accepted belief that the hardness of wheat is associated with and possibly dependent upon its protein content, and it is generally accepted that hard wheats are relatively high in protein. In experiments to obtain direct evidence on the point three methods of testing wheat for hardness have been tried: (1) The method devised by the author, in which hardness is expressed in terms of the crushing-point (*Kans. Agric. Exper. Stat. Bull.*, **167**, 1910, 371-390); (2) The method of Harper and Peters, in which the hardness is determined by means of a piston with a cutting edge, which is pressed down upon the kernel by weights added directly from above (*Kentucky Agric. Exper. Stat. Bull.*, **113**, 1904, 1-12); and (3) the method of Shaw and Gaumnitz, in which the apparatus consists of ordinary pliers, one arm of which is attached to a wooden block, whilst weights are suspended from the extremity of the upper arm until the wheat grain, placed between the jaws of the pliers, is cut (*Calif. Agric. Exper. Stat. Bull.*, **212**, 1911, 315-394). No correlation could be established between the crushing or breaking point of the kernel in grms. and the percentage of protein, nor could any relationship be found between protein content and either specific gravity or volume of the grain.

ORGANIC ANALYSIS.

Volumetric Estimation of the Methoxy Group. J. Troeger and E. Tiebe. (*Arch. Pharm.*, 1920, **258**, 277-287; *J. Chem. Soc.*, 1921, **119**, ii. 135.)—The substance (0.1 gm.), contained in a boat and placed in a tube fitted with a mantle and inspection window, is heated in a stream of dry hydrogen chloride until bubbles of gas are seen to escape from it. Air is displaced before heating and the methyl chloride produced is collected and measured over 30 to 35 per cent. sodium hydroxide solution (which only absorbs 0.3 c.c. of methyl chloride per hour, compared with 7.0 c.c. in the case of water). It is not necessary to correct the volume of gas for the vapour tension of water, but air derived from the acid used to generate the hydrogen chloride must be determined as unabsorbed gas when the gas is transferred to a eudiometer over water, and a correction must also be made for methyl chloride absorbed by the sodium hydroxide. Results obtained with a number of alkaloids were not absolutely correct, but indicated clearly the number of methoxy groups present. The method is inapplicable to substances which volatilise below the temperature of reaction. The Zeisl method is not applicable to substances containing sulphur; the present

method, however, afforded an approximate result with galipenesulphonic acid, but not in the case of another sulphonated compound.

R. G. P.

Detection of Nitrogen in Organic Compounds. C. D. Zenghells. (*Comptes rend.*, 1921, 173, 308-310.)—The proportion of the nitrogen present in an organic compound which is converted into ammonia when the compound is heated with soda lime is increased by previous addition of one part of powdered electrolytic copper to the dry soda lime; any hydrogen sulphide or arsenide which may be formed when sulphur or arsenic is present is fixed by the copper. To detect nitrogen in an organic compound, a small quantity of the latter is mixed in a porcelain crucible with the soda lime and copper mixture, a little of the latter being superposed and the crucible covered with a watch-glass, on the lower surface of which hangs a drop of the author's formol-silver nitrate reagent (*ANALYST*, 1921, 381), whilst a little water on the upper surface acts as a condenser. The crucible is placed on a sand-bath or, better, a quartz plate previously heated strongly, and the heating maintained by means of a small flame; when moisture begins to condense round the reagent, the flame is withdrawn somewhat to one side. In presence of nitrogen in the compound, a silver ring soon forms, and later, possibly a mirror. This reaction is extremely sensitive, even when applied to compounds containing nitrogen united with oxygen. Thus, it is capable of detecting nitrogen in 0.00001 grm. of nitrophenol or 0.00005 grm. of acetoxime, nitrosonaphthol, or picric acid. When the substance to be examined distils or decomposes at a relatively low temperature, use is made of a glass tube 20 cm. in length, closed at one end. Into this are introduced successively a little sugar, soda lime-copper mixture, the latter mixed with the substance, and lastly more soda lime-copper mixture. The tube is closed by a stopper carrying a small tube which is bent at right angles and ends just beneath the watch-glass with the reagent. The closed tube is heated in a horizontal position, the heat being applied first to the soda lime-copper mixture, and last to the sugar, the gases from which expel the ammonia from the tube.

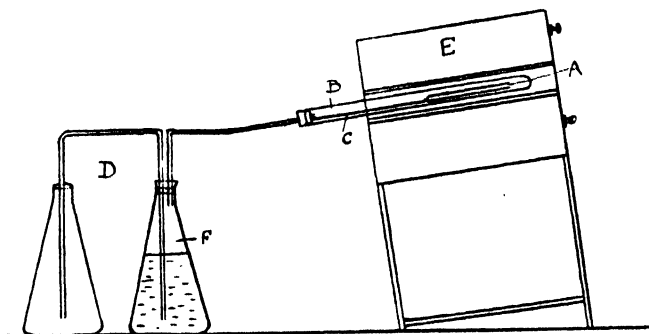
T. H. P.

Decomposition of Potassium Cyanide and Estimation of Formic Acid in the Presence of Hydrocyanic Acid. G. Harker. (*J. Soc. Chem. Ind.*, 1921, 40, 182-185r.)—The yield of gaseous hydrocyanic acid through the action of dilute sulphuric or other acid upon solid potassium cyanide approximates to 30 per cent. of the volume theoretically obtainable, the larger proportion remaining in the solution. The author has carried out experimental work to determine the conditions necessary to ensure the maximum yield of the gas. Warming the reaction mixture leads to decomposition and consequent loss of hydrocyanic acid, but by cooling the mixture and exhausting the apparatus to a pressure of from 16 to 30 mm. of mercury, the loss amounted to 2.9 per cent. only. Hydrolysis of hydrocyanic acid, by heating with either alkali or acid, proceeds slowly; in the former case ammonia and potassium formate are produced quantitatively, but when sulphuric acid is employed the formation of these substances is incomplete. For the estimation of a mixture of formic and hydrocyanic acids the solution is rendered alkaline with potassium hydroxide, and the cyanide estimated as usual by titration with standard

silver nitrate solution. To the solution a second portion of silver nitrate is added exactly equal to that employed for the titration, and the precipitated silver cyanide is removed by filtration. The filtrate is rendered strongly alkaline by the addition of sodium carbonate, gently warmed, and titrated with standard potassium permanganate solution for the estimation of the formic acid present. Experiments made with known quantities of hydrocyanic and formic acids yielded excellent results.

T. J. W.

New Method for Estimating the Volatile Matter yielded by Coals up to Various Temperatures. W. A. Bone and L. Silver. (*J. Chem. Soc.*, 1921, 119, 1145-1152.)—The "American," and the "crucible," and Lessing's methods for estimating volatile matter in coal are open to the objections that the temperature is not accurately regulated, and that air is not excluded. The proposed new method obviates these defects, and indicates precisely the moment when the expulsion of volatile matter ceases. Five grms. of dried coal, which has passed a No. 30 sieve, are placed in the silica tube *A* (12×1.5 cm.), which is supported in its position in the larger silica tube *B* (30×2.5 cm.) by a rod *c*. This "retort" part of the



apparatus is connected with the wash-bottle arrangement *D*, and slowly introduced into the electric furnace *E*, which is inclined at an angle, as indicated, and heated to a temperature of 900°C ., controlled by a pyrometer. The heating is continued till no further gas is evolved, as shown by the water in *D* (usually forty minutes), when the retort system is slowly withdrawn from the furnace, but kept in connection with *D*. The water drawn back into *F* prevents the access of air. The loss in weight gives the volatile matter at 900°C . Coals with a high percentage of volatile matter are introduced into the furnace at 500°C . and gradually heated to 900°C . The results obtained are much more concordant than by other methods, and are in general agreement with the results by the American or crucible tests. H. E. C.

Estimation of Volatile Matter in Graphite. O. L. Shinn. (*J. Ind. Eng. Chem.*, 1921, 13, 633-634.)—The most trustworthy results are obtained by heating the graphite in a platinum boat in a current of pure nitrogen; this method is somewhat tedious, and necessitates the preparation and purification of nitrogen, so the following is proposed as a working method: 1 gm. of the sample is placed in a

platinum crucible provided with a well-fitting lid, the crucible is supported about 10 mm. above a good Meker burner and heated for exactly thirty seconds, then cooled, and re-weighed. The ordinary method of determining volatile matter in coals is unsatisfactory when applied to graphite, owing to oxidation of the latter; at the same time, the short-heating method described above cannot be used for coals and cokes.

W. P. S.

Calcium Chloride Method for the Estimation of Water in Gasoline and in Certain Other Substances. C. W. Clifford. (*J. Ind. Eng. Chem.*, 1921, 13, 628-631.)—Of the various methods recommended for the estimation of water in gasoline that depending on a calculation based on the difference in sp. gr. before and after dehydration is inaccurate, as is also the method in which the water is frozen out by chilling the sample to a low temperature. Dilution with oil dissolving much less water than gasoline is difficult, and the only satisfactory principle is selective absorption by a specific reagent. For this purpose calcium chloride has been found to be the most suitable absorbent. In this method a current of air, dried previously by calcium chloride, is passed through the sample at the rate of 5 to 15 litres per hour for one to two hours; if a part of the water has settled out in the sample it must be kept in suspension by frequent shaking or by the current of air itself. The moisture abstracted from the sample is absorbed by passing the air through two calcium chloride tubes. If the sample consists of a volatile liquid, such as gasoline, benzene, chloroform, or carbon disulphide, a current of dry air must be passed subsequently through the calcium chloride tubes for one hour in order to expel vapour from the tubes before these are weighed. The method is also applicable to sugar, certain pigments, flowers of sulphur, and rubber stock, but cannot be used in the case of acetone, pyridine, ethyl alcohol, and glycerol.

W. P. S.

Estimation of Water in Transformer Oils. E. Rengade and J. Clostre. (*Comptes rend.*, 1921, 173, 311-313.)—None of the methods previously described for this estimation giving satisfactory results, the authors recommend the following procedure: 200 c.c. of the oil are placed in a short-necked flask, closed by a three-holed stopper, carrying a thermometer and entry and exit tubes for passing air through the oil. The exit tube is bent downwards at an acute angle, and connected, by means of a small rubber stopper, with a condensing tube, consisting of an inclined branch, ending in a U-tube immersed in a d'Arsonval vessel; the orifice of the condenser may thus be brought within a very small distance of the stopper of the flask. The whole is maintained at 80° C. by means of an oil-bath, which surrounds the flask up to the neck and is continued above as an air-bath of asbestos card, through which pass the thermometer and the tubes. Thus no vapour condenses before reaching the condenser, which is cooled by either liquid air or solid carbon dioxide. Air, dried by means of sulphuric acid or phosphoric anhydride, is passed through the heated oil at the rate of 20 litres per hour. The increase in weight of the condenser during the first fifteen minutes represents solely water, and gives a minimal value for the water content; probably a more accurate value is obtained in thirty minutes,

the small amount of oil then condensing being counterbalanced by the water found to be liberated during the third period of fifteen minutes. T. H. P.

True Oxygen Absorption of Drying Oils. S. Coffey. (*J. Chem. Soc.*, 1921, 119, 1152-1161.)—The so-called "oxygen value" of drying oils calculated from the maximum increase in weight is really only an apparent value, since, owing to the simultaneous evolution of volatile decomposition products, the real oxygen absorption is considerably greater than the percentage increment in weight. For the estimation of the true value a modified form of Genthe's apparatus (*Zeitsch. angew. Chem.*, 1906, 19, 2087) was used. A filter-paper (11 cm. in diameter) was saturated with a "standard" solution of linseed-oil in petroleum spirit (b.pt. 40° to 60° C.), and suspended in the apparatus, which was then closed, completely filled with hydrogen, and placed in a thermostat at 100° C. When the temperature of the film of oil was constant, the apparatus was rapidly filled with oxygen, and manometer readings were taken at intervals. The volume of the apparatus was determined by measuring the volume of water required to fill it, corrections being applied for the volume of absorbents and manometer leads. Broken pumice, soaked in sulphuric acid, and a stick of sodium hydroxide wrapped in copper gauze were used as absorbents. The oxygen absorption of pure linseed oil (iodine value, 184.1) thus determined ranged from 28.6 to 29.2 per cent., and that of the fatty acids of the same oil from 30.05 to 30.20 per cent. In most cases the error in the estimation is within 1 per cent. The true oxygen absorption is much higher than the values hitherto recorded, and, unlike the apparent oxygen absorption, is a definite quantity for a given oil. The ratio of oxygen absorption to iodine absorption is 0.156. The oxidation is thus not a simple molecular autoxidation, for the oxygen absorption is about 25 per cent. higher than it would be on this assumption, which requires an oxygen absorption of 22.7 per cent. This leaves an *excess* oxygen absorption of 6.0 per cent., which may give rise to the volatile acid products, and it is possible that a relationship might be traced between the amount of oxygen in these products and the *excess* oxygen absorption.

Linolenic and Hexabromostearic Acids. S. Coffey. (*J. Chem. Soc.*, 1921, 119, 1306-1310.)—Hehner and Mitchell (*ANALYST*, 1898, 23, 310) prepared linolenic acid from purified linolenic acid hexabromide (m.pt. 180° to 181° C.) and found it to have sp. gr. 0.9288 at 15.5°/15.5° C. On re-bromination it yielded only 46 to 50 per cent. of the theoretical amount of hexabromide. Erdmann and Bedford (*Ber.*, 1909, 42, 1324) showed that this was due to the fact that linolenic acid obtained by reducing linolenic acid hexabromide with zinc consists of a mixture of α - and β -linolenic acids, only the α -modification being of natural occurrence. More recently Erdmann (*Zeitsch. physiol. Chem.*, 1911, 74, 179) claims to have prepared pure α -linolenic acid by fractional crystallisation from alcohol of the zinc salts of the liquid fatty acids of linseed oil. The zinc salt $(C_{18}H_{33}O_2)_2Zn, \frac{1}{2}ZnO$, melted at 72° to 73° C., and the yield was nearly theoretical, corresponding with 18 per cent. of linolenic acid in the liquid fatty acid of linseed oil. The author, however, has been unable to isolate pure zinc linolenate, the differences in the solubility of zinc linolenate and

zinc linolate being not nearly so pronounced as stated by Erdmann; in fact, the most soluble product was a mixture consisting mainly of zinc linolate. The statements of Erdmann and Bedford (*loc. cit.*) concerning the mixtures of α - and β -acids obtained from the hexabromide have been confirmed. Pure alkaloidal salts of hexabromostearic acid have been prepared. The strychnine salt crystallises in flat microscopic rhombs (m.-pt. 185° C.), the morphine salts in rhombs (m.-pt. 181° to 182° C.), and the narcotine salt melts at 184° C.

Characterisation of Amylase Solutions. H. von Euler and O. Svanberg. *Zeitsch. physiol. Chem.*, 1921, **112**, 193-230; *J. Chem. Soc.*, 1921, **120**, ii. 528.)—After reviewing the literature on the subject, a new "absolute" measure for the activity of amylase preparations is advocated which is analogous to the author's method for invertase (*Zeitsch. physiol. Chem.*, 1919, **106**, 201). The reaction velocity is measured with soluble starch (Lintner), previously boiled, at concentration of 0.72 to 2.8 per cent. and temperature 37° C. The P_H value should be approximately 5, and the enzyme concentration such that the constant (*Sf.*) is between 0.004 and 0.08. The constant *Sf.* then equals ($K \times$ maltose/enzyme preparation); the concentrations are expressed in grms. Here *K* is the velocity constant of the unimolecular reaction by which the hydrolysis proceeds to the formation of a maximum quantity of maltose. Experiment shows that 1,000 Lintner units = 26 *Sf.*, and 1,000 of Sherman's units = 38.5 *Sf.*

H. E. C.

Significance of "Lignin" Colour Reactions. E. C. Crocker. (*J. Ind. Eng. Chem.*, 1921, **13**, 625-627.)—The colorations obtained with phloroglucinol, *p*-nitroaniline, etc., are due to the presence of traces of an aldehyde which usually, if not always, accompanies lignin; it appears that there is one single aldehyde in wood, or that one aldehyde predominates, and that this aldehyde is probably coniferyl aldehyde. Vanillin or furfural, if present, constitutes only a small fraction of the aldehyde content of wood. Several non-aldehydic substances which yield reactions similar to those of "lignin" are shown to contain traces of aldehydes which are responsible for the colour formation; in the case of clove oil and sassafras oil, the aldehyde seems to be identical with that of wood. The Maule reaction (red coloration when wood is treated successively with permanganate, dilute hydrochloric acid, and ammonia) is essentially a chlorination of some wood constituent, followed by alkaline treatment; the red coloration is obtained generally in the case of deciduous woods, an indefinite brown coloration being given by coniferous woods, and the reaction may be of value in differentiating these woods.

W. P. S.

Estimation of Cresol by the Phenol Reagent of Folin and Denis. R. M. Chapin. (*J. Biol. Chem.*, 1921, **47**, 309-314.)—Factors affecting the accuracy of Folin and Denis's method of estimating phenols (*J. Biol. Chem.*, 1915, **22**, 305) are discussed, and the use of the following procedure and factors is recommended: Three c.c. of the silico-tungstic acid reagent made up by Wu's method (*J. Biol. Chem.*, 1920, **43**, 189) are added to a quantity of liquid containing about 0.5 mgrm. of phenol to produce the maximum colour, and the mixture diluted; then 5 grms. of

sodium bicarbonate are added and the mixture allowed to stand for thirty minutes before the addition of 5 c.c. of 10 per cent. sodium sulphite solution; a further fifteen minutes should be allowed before colorimetric comparison with the standard to allow of full development of the colour. When phenol is employed as a standard in the estimation of cresols the following are the factors equivalent to 1 part of phenol: Ortho-cresol, 1.19; *m*-cresol, 1.09; *p*-cresol, 1.22; or, for commercial cresol (85 : 40 : 25), the factor is 1.16. For the determination of phenolic preservatives in sera, 1 c.c. of the sample is treated in a 300 c.c. flask with 125 c.c. of water, 4 c.c. of 1 : 3 sulphuric acid, 4 c.c. of 12 per cent. silico-tungstic acid, and fragments of hot pumice. The contents are distilled into a 200 c.c. flask; when 100 c.c. have passed over, a further 100 c.c. of water are added to the contents in the distillation flask, and the distillation continued till 200 c.c. have passed over. The phenol in the distillate is then determined in 25 or 50 c.c., depending on whether phenol or cresol is the preservative present.

H. E. C.

Volumetric Estimation of Aminonaphthol-mono- and di-sulphonic Acids. G. R. Levi. (*Giorn. Chim. Ind. Appl.*, 1921, 3, 297-302.)—Aminonaphthol-sulphonic acids may be titrated with sodium nitrite in acid solution, and also with a diazo-compound, such as diazobenzene or diazotised *p*-nitrobenzene, and the author has determined the nitrite number and the diazo number for the following acids of importance in the azo-dye industry: *M*-acid ($\text{NH}_2 : \text{OH} : \text{SO}_3\text{H} = 1 : 5 : 7$), *S*-acid ($1 : 8 : 4$), γ -acid ($2 : 8 : 6$), *J*-acid ($2 : 5 : 7$), *R*-acid ($2 : 3 : 6$), 2-Sacid ($\text{NH}_2 : \text{OH} : \text{SO}_3\text{H} : \text{SO}_3\text{H} = 1 : 8 : 2 : 4$), *H*-acid ($1 : 8 : 3 : 6$), *K*-acid ($1 : 8 : 4 : 6$), and 2 *R*-acid ($2 : 8 : 3 : 6$). The nitrite number is determined by running $\frac{N}{2}$ -sodium nitrite solution slowly into a fresh solution or suspension of the acid containing mineral acid. Considerable excess of the latter does not increase the velocity of diazotisation appreciably, but this is influenced by rise of temperature, so that it is advantageous to carry out the first part of the diazotisation at 10° to 12° C., and then to raise the temperature to 25° or even 30° C. If the sulphonic acid be of good quality, the difference between the nitrite and diazo numbers should not exceed 0.5 or 0.2 per cent. for dry and pasty products respectively.

T. H. P.

Analysis of Wool Waste or Refuse. A. Demolon. (*Ann. Chim. anal.*, 1921, 3, 244-246.)—Discrepancies in the estimation of total nitrogen in wool waste are probably due to difficulty of obtaining a representative sample for the estimation, and to insufficient heating during the digestion with sulphuric acid in the Kjeldahl method. It is recommended that about 10 grms. of the material be heated gently in a weighed porcelain basin with 15 c.c. of sulphuric acid until a fluid mass is formed; calcium sulphate is then added gradually, with stirring, so as to produce a dry powder and increase the total weight to 80 grms. Eight grms. of this powder (equal to 1 gm. of original material) are then taken for the estimation of the total nitrogen. The heating with sulphuric acid (Kjeldahl method) should be continued for two hours after a clear solution has been obtained.

W. P. S.

INORGANIC ANALYSIS.

Note on the Gasometric Determination of Nitrogen. R. L. Stehle. (*J. Biol. Chem.*, 1921, **47**, 11.)—In the process previously described (*ANALYST*, 1921, 110), the absorption of oxygen by pyrogallate is unnecessary if copper sulphate be omitted from the Kjeldahl digestion, as then only nitrogen is liberated on treatment with the sodium hypobromite. H. E. C.

Estimation of Metals as Sulphides. L. Moser and A. Schattner. (*Chem. Zeit.*, 1921, **45**, 758-759.)—The quantitative estimation of metals in their compounds is effected by precipitating them as sulphide, which is then obtained in a form suitable for weighing by heating in a current of hydrogen sulphide. The method gives excellent results with zinc, cadmium, manganese, and silver. In the case of iron, however, it is necessary to heat the sulphide in presence of both hydrogen sulphide and hydrogen in order to avoid the formation of higher sulphides. Zinc sulphide is obtained in a stable amorphous or partially crystalline form by heating it for thirty to forty-five minutes in the current of gas at 570° to 590° C., the temperature being controlled by means of a thermo-element placed near the crucible. Zinc oxide, either dried or ignited, may be quantitatively converted into zinc sulphide by heating it to redness in hydrogen sulphide. The same method is also applicable to cadmium oxide or carbonate, to manganese sulphate, or the oxides Mn_2O_3 and MnO_2 , and to silver chloride, nitrate or oxide, the respective sulphides being quantitatively obtained in each case. W. J. W.

Zinc Purpurate as a Reagent for Mercury Salts. G. Denigès. (*Bull. Soc. Pharm. Bordeaux*, 1921; *Ann. Chim. anal.*, 1921, **3**, 252-253.)—The reagent is prepared as follows: Two grms. of uric acid are heated with 2 grms. of nitric acid until solution is complete; 2 c.c. of water are then added, the mixture again heated and diluted with water to 100 c.c.; 10 c.c. of this solution are boiled for five minutes with the addition of 2 grms. of zinc. The solution becomes orange-yellow owing to the formation of zinc purpurate. A characteristic pink-coloured precipitate of mercury purpurate is obtained when a solution containing a trace of mercury (*e.g.*, 0.4 mgrm. per c.c.) is treated with a few drops of the reagent followed by a small quantity of sodium acetate solution. Silver salts, under the same conditions, yield a violet-coloured precipitate. W. P. S.

Compounds of Halogenated Derivatives of Mercury and Thallium. [Separation of Mercury from Thallium.] J. Barlot and J. Pernot. (*Comptes rend.*, 1921, **173**, 232-234.)—The separation of mercury from thallium involved in the analysis of compounds of the type, $HgCl_2$, $TlCl$, may be effected by taking advantage of the fact that thallos sulphide, but not mercuric sulphide, dissolves easily in dilute mineral acid. The double salt is transformed into a mixture of nitrates by treatment with nitric acid, the liquid being approximately neutralised with ammonia and the two sulphides precipitated by excess of ammonium hydrosulphide. After filtration, the precipitate is treated with 10 per cent. nitric acid, which dissolves the thallos

sulphide; the liquid is boiled to expel hydrogen sulphide, neutralised exactly with ammonia, and treated with potassium iodide, the thallium being weighed as thalious iodide; the solubility of the latter is diminished by the presence of excess of potassium iodide.

T. H. P.

Quantitative Separation of Tin and Antimony. M. Mouret and M. J. Barlot. (*Bull. Soc. Chim.*, 1921, 29, 743-745.)—The following modification of Plato's method is recommended: The alloy is dissolved in *aqua regia*, the solution evaporated almost to dryness, and the residue dissolved in hot concentrated hydrochloric acid. An equal volume of water is added, then 50 c.c. of a solution containing not less than 4 grms. of crystalline orthophosphoric acid, and the solution is heated to 80° to 90° C., and a current of hydrogen sulphide is passed through it for thirty minutes. The antimony is completely precipitated as sulphide, and, after standing fifteen minutes, the precipitate may be filtered on a Gooch filter of double thickness, washed with hydrogen sulphide water, and dried in a current of carbon dioxide. Alternatively, the antimony sulphide may be electrolysed in a solution containing hydroxylamine hydrochloride, a current of 0.5 ampère at from 2 to 2.5 volts being used. The phosphoric acid solution containing the tin is heated to eliminate hydrogen sulphide, and a portion, containing not more than 0.04 gm. of tin, is treated with "cupferron," and continually stirred until the precipitate is flocculated. After filtration, the residue is washed with cold water, dried at a low temperature in a vacuum desiccator, ignited, and weighed. The method described is rapid, and the results obtained are in close agreement with the quantities of each metal present in an alloy.

T. J. W.

The Dimethylglyoxime Reactions of Iron and Cobalt. W. Vaubel. (*Zeitsch. öffentl. Chem.*, 1921, 27, 163-164.)—Ferrous salts yield an intense Bordeaux red coloration when treated with dimethylglyoxime solution and ammonia; the coloration is distinct from the red colour and precipitate given by nickel salts. Ferric salts also give the reaction, but only after reduction by a trace of ammonium sulphide, added after the dimethylglyoxime. If 20 c.c. of a cobalt salt solution, containing 2 mgrms. of cobalt, be mixed with 20 c.c. of dimethylglyoxime solution, and the mixture diluted to 50 c.c. and treated with 10 drops of ammonium sulphide solution and 10 drops of ammonia, a blue-violet to deep red coloration is obtained. This reaction may be used for the detection of cobalt in the filtrate from the precipitate obtained with nickel.

W. P. S.

Estimation of Vanadium in Steels and Iron Alloys. L. Rolla and M. Nuti. (*Giorn. Chim. Ind. Applic.*, 1921, 3, 287.)—Vanadium is quantitatively precipitated from a solution containing about 1 per cent. of free hydrochloric or sulphuric acid by cold 4 per cent. "cupferron" solution; the precipitate is filtered off, washed with 2 per cent. hydrochloric or sulphuric acid solution containing 2 per cent. of the 4 per cent. cupferron solution, dried in an oven at about 70° C., burned and calcined to constant weight, and weighed as V_2O_5 . The separation of iron and chromium from vanadium may be effected by means of boiling sodium hydroxide solution, the

first two elements being eliminated as hydroxides; in order to avoid the formation of a voluminous precipitate, the bulk of the iron may be removed previously by means of ether, as in Campagne's method. Tungsten may be eliminated as tungstic anhydride in the ordinary way. Since vanadium may be separated also from phosphorus in a solution containing hydrochloric or sulphuric acid, the method may be applied to ferrovanadium: About 0.5 gm. is disaggregated in an iron crucible by treatment with six times its weight of sodium peroxide, the mass being taken up in water, acidified, and freed from silica; the filtrate, free from iron, is treated with cupferron as described above. In the case of steels sodium peroxide cannot be employed, since the large quantity of alkali salts formed would necessitate such dilution that the resulting solution would not contain a measurable amount of vanadium. Use is made, therefore, of the solution in hydrochloric acid, the silica and tungsten being eliminated by filtration, and the iron by means of ether. The liquid containing the chromium and vanadium is then boiled with sodium carbonate and filtered, the filtrate being treated either with lead acetate or with acetic acid and lead nitrate. The lead vanadate thus precipitated is collected and dissolved in dilute hydrochloric acid, the solution being evaporated with concentrated sulphuric acid, the residue dissolved in water, and the solution filtered and treated with "cupferron" to estimate the vanadium.

T. H. P.

Detection and Estimation of Potassium as Picrate. S. Minovici and A. Jonescu. (*Bull. Soc. Chim. România*, 1921, 3, 25-33; *J. Chem. Soc.*, 1921, 120, ii., 520.)—Potassium is precipitated quantitatively in the form of yellow crystals by a saturated solution of picric acid in 95 per cent. alcohol containing 5 per cent. of glycerol; the crystals are collected, washed with ether, dried under reduced pressure, and weighed. Alternatively, the precipitate may be dissolved and titrated with quinine hydrogen sulphate, which in dilute solution precipitates picric acid completely. The method is adaptable to the microchemical estimation of potassium, being sensitive to 0.01 mgrm. Sodium and ammonium salts do not yield crystals.

H. E. C.

Estimation of Bromine in Saline Waters. P. Lebeau and M. Picon. (*Bull. Soc. Chim.*, 1921, 29, 739-743.)—The following modification of L. Figuier's method (*Ann. Chim. Phys.*, 1851, 33, 303) is described: In order to avoid the addition of excess of chlorine in the estimation a preliminary test is made by adding increasing amounts of an aqueous solution of chlorine containing 0.5 gm. per litre to a series of tubes each containing 1 c.c. of the water under examination. The volume of chlorine solution required to cause a faint decrease in the coloration due to the liberated bromine will be approximately double the volume required to displace the whole of the bromine present. In the actual estimation a volume of water, containing approximately 100 mgrms. of bromine, is introduced into a separating funnel, and 5 c.c. of 10 per cent. hydrochloric acid are run in, followed by a quantity of 0.5 per cent. chlorine solution, calculated from the preliminary experiment. Fifteen c.c. of chloroform are then added, and the liquid gently agitated to avoid emulsification, and, after separation, run into a flask containing 10 c.c. of 10 per cent. potassium

iodide solution. The saline solution is washed with further portions of chloroform until these remain colourless, when from 0.3 to 0.5 c.c. of chlorine solution is added to the contents of the separating funnel, and the chloroform extraction repeated until bromine ceases to be liberated. The chloroform extracts are shaken with the potassium iodide solution, and the whole titrated with $\frac{N}{10}$ sodium thiosulphate solution. The quantity of saline solution used should not exceed 25 c.c., and, if containing only a small quantity of bromine, should be concentrated by evaporation. The results obtained are accurate within 1 per cent. of the total bromine present.

T. J. W.

Analysis of Fibro-Cement. B. J. Smart and P. C. Pecover. (*J. Soc. Chem. Ind.*, 1921, 40, 185-186r.)—Abnormal values being obtained in the determination of the lime value of the cement contained in an asbestos cement by extraction with 20 per cent. hydrochloric acid, investigation by the authors showed that the residual asbestos had adsorbed silica from the solution, and that this silica was capable of removal by extraction of the asbestos with alkali. Five grms. of the coarsely ground material were covered with 200 c.c. of water in a porcelain basin, and 40 c.c. of hydrochloric acid gradually added, with constant stirring, after which the mixture was allowed to stand for twenty-four hours. The whole was filtered, and the residue washed with 20 per cent. hydrochloric acid until the washings were colourless, after which it was dried, ignited, and weighed. The filtrate and washings were diluted to a known volume, and an aliquot part used for the estimation of calcium, silica, etc. From the known calcium-silica ratio of the cement used the proportion of silica adsorbed was estimated, this being confirmed by extraction of the residual asbestos with caustic alkali and estimation of the silica dissolved in the solution. By this method the asbestos found in three estimations in a cement prepared in the laboratory and containing 16.7 per cent. amounted to 16.6, 16.4, and 16.6 per cent.

T. J. W.

ERRATA.

Production and Testing of Zirconia (ANALYST, 1921, 344).—For "sulphite," line 19, read "sulphur trioxide," and for "arsenious acid," line 24, read "arsenic."

PHYSICAL METHODS, APPARATUS, ETC.

Apparatus for the Industrial Analysis of Gases. G. Ardoyer. (*Comptes rend.*, 1921, 173, 237-238.)—This apparatus consists of a vertical measuring tube, which is divided into tenths of a c.c., is surrounded by a water-jacket, and is connected at its lower extremity with a levelling tube containing salt water; the upper end is fused to a short, narrow, horizontal tube carrying a three-way cock, from which the graduation commences, and, beyond the cock, a small funnel, the third way being left free. The gas is introduced into the measuring vessel through this third way, which is connected in turn with the various Orsat pipettes, after the volume of the gas has been measured. An explosion chamber or a combustion tube charged with metallised asbestos and heated with a small flame may also be connected. The apparatus is simple, strong, and portable, and yields accurate results.

T. H. P.

THE INSTITUTE OF BREWING RESEARCH SCHEME.

THIS scheme, organised and financed by the Institute of Brewing for the solution of problems connected with the brewing and allied industries, has given an impetus to investigation in various branches of science which have been somewhat neglected during recent years. Systematic work in connection with hops is now in progress. The culture of new varieties, manuring experiments, and chemical analysis of hops are conducted under a grant from the Institute and the Kent County Council at the East Malling Fruit Research Station and at Wye Agricultural College, whilst four experimental drying kilns have been recently completed for the Institute at Paddock Wood.

The estimation of the resin content of hops was carried out as follows: Whole hops were soaked in petroleum spirit for twelve hours at the ordinary temperature, and digested on a water-bath for a further five hours. The mixture was filtered, the filtrate diluted to a definite volume, an aliquot portion dried, and the residue of soft resins was weighed. The total (hard and soft) resins were estimated in a similar manner by substituting ether for the petroleum spirit previously used. The total resins in twenty-two varieties over a period of four years ranged from 10.6 to 22.1 per cent., whilst the soft resins varied between 6.7 and 11.6 per cent. The percentage of hard resin present appears to be characteristic for a particular variety of hop.

Manuring experiments in the field will be conducted upon separate plots, with the following variations in treatment: (a) Unmanured, (b) complete mineral manure, (c) complete mineral manure with and without (i.) organic manure (dung), (ii.) nitrogen, (iii.) phosphorus, and (iv.) potassium.

Chemical investigations are being carried out by Dr. Pyman in the College of Technology, Manchester, with the object of isolating and estimating the constituent or constituents to which the hop owes its antiseptic or preservative qualities.

Research upon timber used in the construction of casks is in progress from the botanical, biological, and chemical standpoints. American white oak, owing to the presence of starch in the heartwood, is liable to lead to the development of foreign organisms and the deterioration of beer, whilst American red oak is unsuitable for use in barrels, owing to its permeable nature. The chemical investigation consists of: (a) The estimation of variations in extract yielded to various solvents; (b) the conditions under which such extracts are taken up by beer; and (c) the determination of whether such extracts are capable of communicating undesirable flavours to beer. Extracts of various woods obtained up to the present by the use of benzene, alcohol, and water, show large variations in quality.

T. J. W.



STATUTORY RULES AND ORDERS, 1921, No. 1305.**FOOD CONTROL.**

THE SALE OF FOOD ORDER, 1921, DATED AUGUST 16, 1921, MADE BY THE BOARD OF TRADE UNDER THE MINISTRY OF FOOD (CONTINUANCE) ACT, 1920 (10 AND 11 GEO. V., c. 47, AND THE MINISTRY OF FOOD (CESSATION) ORDER, 1921.

IN exercise of the powers conferred upon them by the Ministry of Food (Continuance) Act, 1920, and the Ministry of Food (Cessation) Order, 1921, and of all other powers enabling them in that behalf, the Board of Trade hereby order as follows:

PART I. : BREAD.—1. All bread (other than bread sold for consumption on the premises of the seller) shall be sold by weight and not otherwise. 2. No loaf of bread shall be sold unless its weight be 1 pound or an even number of pounds. 3. No roll of bread exceeding 2 ounces in weight shall be sold. 4. Bread which may not under the foregoing provisions of this Order be lawfully sold shall not be offered or exposed or carried for sale or delivered under a contract for sale. 5. Any person authorised by a local authority or any Inspector of Weights and Measures may require any person offering or exposing or carrying any bread for sale or delivering any bread under a contract for sale to weigh such bread in the presence of such person or Inspector or permit such person or Inspector to weigh such bread.

PART II. : TEA.—6. (a) All tea sold by retail, whether contained in a package or not, shall be sold by net weight, and in ounces or pounds or in multiple of ounces or pounds and not otherwise, provided that this shall not apply to tea sold in quantity of less than 2 ounces.

(b) A person shall not place on any package of tea intended for sale or on any wrapper, band, or label affixed thereto, any statement as to weight, or sell or offer or have in his possession for sale by retail any package bearing on the package or on any wrapper, band, or label affixed thereto any statement as to weight, unless in either case such statement is a true statement of the net weight of the tea contained in such package.

PART III. : LABELLING OF IMPORTED PRODUCE.—7. (a) A person shall not expose for sale by retail any imported meat or any imported bacon, ham, or lard unless the article bears at the time of exposure for sale a label with the word "imported" or with a word or words disclosing the country of origin of the article clearly printed thereon, so as to be easily readable by the customers:

Provided that where only imported meat or only imported bacon, ham, or lard is exposed for sale on any slab, rail, or counter it shall be sufficient compliance with the requirements of this clause if the slab, rail, or counter bears in a conspicuous position such a label:

Provided also that where all the meat for the time being on sale in any premises is imported meat, it shall be a sufficient compliance with the requirements of this clause if there is exhibited on the premises in a conspicuous position, and so as to be easily readable by the customers, a notice stating that imported meat only is on sale:

Provided also that where pieces of home-killed and imported meat, not exceeding in any case 1 pound in weight, are exposed for sale on a slab, tray, or counter, the foregoing provisions of this clause shall not apply to the imported meat so exposed, provided that the slab, tray, or counter bears in a conspicuous position a notice containing the words "mixed home-killed and imported meat" clearly printed thereon.

(b) On the occasion of a sale, other than a sale by retail, of any imported bacon, ham, or lard, the seller shall give to the buyer an invoice accurately stating: (i.) that the bacon, ham, or lard is imported, and (ii.) in the case of bacon or ham, the country of origin.

(c) For the purposes of this clause: "Meat" shall include beef, mutton, lamb, pork, and veal, but shall not include bacon or ham, or cooked, canned, or potted meat, sausages or offals. "Bacon" shall include shoulders and picnics, but shall not include pickled pork or cured pigs' heads. "Lard" shall not include neutral lard or compound lard. "Imported" shall mean with respect to any bacon, ham, or lard, cured or manufactured outside the United Kingdom, or cured or manufactured in the United Kingdom from pigs raised outside the United Kingdom. Lard which contains any imported lard shall be deemed to be imported lard.

8. A person shall not sell or offer or expose for sale, whether by wholesale or retail, as fresh eggs or new-laid eggs or under any description of which the words "fresh" or "new-laid" form part, any eggs which have been imported into the United Kingdom, unless the description also includes the word "imported" or a word or words disclosing the country of origin.

PART IV. : JAM.—9. A person shall not sell or offer or expose for sale any jam unless the same complies with the following provisions: (a) The water-soluble extract of jam shall not be less than 65 per cent. of the jam. (b) Not more than 10 per cent. of the jam measured by weight shall consist of added fruit juice. (c) Where more than one variety of fruit or vegetable is used in the making of a jam [other than in the form of added fruit juice not exceeding the quantity specified in sub-clause (b)] each such variety must be mentioned in the description, but so that where any fruit or fruits contained in such jam is less than 25 per cent. of the total fruit content, the name of such fruit or fruits shall be prefaced in the description by the words "Flavoured with" in such form as to be easily readable by the buyer. (d) The provisions of sub-clause (c) shall not apply to jam sold under the description of "Mixed Jam" or "Mixed Fruit Jam." (e) Notwithstanding the provisions of sub-clause (c), jam made from rhubarb and preserved ginger may be sold under the description "Rhubarb and Preserved Ginger," provided that the quantity of preserved ginger contained in such jam is not less than 20 per cent. of the total fruit content.

10. Without prejudice to the provisions of Clause 9, a person shall not sell or offer or expose for sale any article under the description of marmalade or under any description of which the word "marmalade" forms part, unless (a) only citrous fruits, citrous fruit juices, and sugar or other sweetening substances have been used in the making thereof; or (b) each variety of fruit or vegetable used in the making thereof is mentioned in the description.

11. (a) A person shall not sell or offer or expose for sale or deliver pursuant to any contract of sale, whether by wholesale or by retail, any jam in a container unless such container bears (i.) the name and address of the manufacturer of the jam, or in the case of imported jam, a word or words disclosing the country of origin; provided that this sub-clause shall not apply to a sale of jam where the jam has been manufactured for a retailer under a contract to manufacture made between the manufacturer and the retailer and is sold by the retailer as manufactured for himself, but in any such case, in any proceedings against the retailer in respect of a sale or offer or exposure for sale of the jam, the provisions of Clause 21 of this Order shall not be available as a defence; (ii.) except where the jam is packed in usual containers with a net content of 1 pound, 2 pounds, 3 pounds, or 7 pounds, the guaranteed net weight of the contents.

(b) Notwithstanding the preceding provisions of this clause, jam imported from any of the British Dominions beyond the seas may be sold in a container which does not bear the guaranteed net weight of the contents, provided that in the case of a wholesale sale the seller shall give to the buyer an accurate statement in writing showing the net weight of the contents of the container, and that in the case of a retail sale the seller shall keep posted in a conspicuous position, so as to be easily readable by all customers throughout the time during which the jam

is being sold or exposed for sale, a notice showing in plain words or figures the net weight of the contents of the container, in accordance with the statement given to him by the wholesaler.

(c) A person shall not make or knowingly connive at the making of any false statement as to the matters set out in this clause, or alter or deface any label or other writing regarding such matters.

12. Every person selling jam shall produce all books of account, records, and invoices to any person authorised by the local authority to inspect the same, so far as necessary for the purpose of showing whether or not he is complying with the provisions of this Part of this Order as respects content, weight, description, and labelling.

13. For the purposes of this Part of this Order the expression "Jam" shall include jelly, conserve, and marmalade.

PART V. : FATS.—14. A person shall not sell or offer or expose for sale as dripping any substance unless such substance shall have been manufactured by a process other than the acid process from raw-beef fat or raw-mutton fat or beef or mutton bones, and does not contain more than 2 per cent. of free fatty acids or more than 1 per cent. in all of water and substances other than fat.

15. A person shall not sell or offer or expose for sale any substance (except lard, neutral lard, oleo oil, beef and mutton stearine and Premier Jus) manufactured from raw-beef fat or raw-mutton fat or beef or mutton bones, or from any other animal fat, which does not comply with the requirements of the preceding clause, otherwise than under the name and by the description of "Technical Tallow."

16. A person shall not sell or offer or expose for sale as an edible fat any mixture or compound (other than butter, margarine, or dripping) manufactured wholly or partly from oils or fats, which contains more than 0.5 per cent. of free fatty acids or more than 0.5 per cent. in all of water and substances other than oil or fat.

17. A person shall not manufacture or sell or offer or expose for sale any margarine which does not contain at least 80 per cent. of oil and fat.

PART VI. : GENERAL.—18. (a) Every local authority is hereby authorised to execute and enforce the provisions of this Order within their area, and except in Scotland to institute proceedings for any offences against this Order. (b) For the purposes of Parts I. and II. of this Order the local authority shall be the local authority for the purposes of the Weights and Measures Acts, 1878 to 1919. (c) For the purposes of Parts III., IV., and V. of this Order the local authority shall be any local authority authorised to appoint an analyst for the purposes of the Sale of Food and Drugs Acts, 1875 to 1907.

19. A person authorised in that behalf by a local authority to procure for analysis samples of any article to which Part IV. or Part V. of this Order applies shall have all the powers of procuring samples conferred by the Sale of Food and Drugs Acts, 1875 to 1899, and a person selling or exposing for sale any such article shall, on tender of the price for the quantity which he shall reasonably require for the purpose of analysis, sell the same to such authorised person accordingly.

20. In any proceedings in respect of an infringement of this Order the production of the certificate of the principal chemist of the Government laboratories, or of an analyst appointed under the Sale of Food and Drugs Acts, 1875 to 1907, shall be sufficient evidence of the facts therein stated unless the defendant shall require that the person who made the analysis shall be called as a witness. The certificate shall, so far as circumstances permit, be in the form required by the Sale of Food and Drugs Act, 1875.

21. (i.) If in any proceedings for an infringement of any of the provisions of this Order (other than the provisions of Part I. or Part III.) the defendant proves (a) (1) in the case of proceedings under Part II., that he purchased the article in the package in which he sold the same or offered or exposed it for sale, and with a written warranty of the net weight of the article con-

tained in the package, or with a statement on the package of such net weight, (2) in the case of proceedings under Part IV., that he sold or offered or exposed for sale the article in the container in which he bought it, (3) in the case of proceedings under Part V., that he bought the article with a written warranty as to such of the matters referred to in Part V., in respect of which it is proved that an offence has been committed; (b) in any case that he had no reason to believe that the article did not, as respects content, weight, description, or labelling (as the case may be), comply with the provisions of this Order; (c) that he has given due notice to the prosecutor that he intends to rely upon the provisions of this clause. Such person shall be entitled to be discharged from the prosecution.

(ii.) The provisions of sub-sections (3) and (4) of Section 20 of the Sale of Food and Drugs Act, 1899, shall apply as nearly as may be to proceedings under this clause in the same way as they apply to proceedings under the Sale of Food and Drugs Acts.

22. For the purposes of this Order percentages shall be calculated by weight.

23. Infringements of this Order are summary offences under the Ministry of Food (Continuance) Act, 1920.

24. The Orders mentioned in the schedule to this Order are hereby revoked, but without prejudice to any proceedings in respect of any contravention thereof.

25. (a) This Order may be cited as the Sale of Food Order, 1921, and shall come into force on September 1, 1921. (b) This Order shall not apply to Ireland. (c) The Interpretation Act, 1889, applies to the interpretation of this Order as it applies to the interpretation of an Act of Parliament.

By Order of the Board of Trade,

FRANK H. COLLIER,

Secretary to the Food Department.

August 16, 1921.

THE SCHEDULE.—S.R. and O., 1918, No. 547, the Bread Order, 1918; S.R. and O., 1917, No. 818, the Tea (Net Weight) Order, 1917; S.R. and O., 1921, No. 408, the Bacon, Ham, and Lard (Sales) Order, 1921; S.R. and O., 1919, No. 1783, and 1920, No. 858, the Imported Meat (Labelling) Order (No. 2), 1919, as amended; S.R. and O., 1920, No. 2408, the Eggs (Description on Sale) Order, 1920; S.R. and O., 1920, No. 1512, and 1921, No. 215, the Jam (Sales) Order, 1920, as amended; S.R. and O., 1919, No. 511, the Dripping (Standard of Quality) Order, 1919; S.R. and O., 1919, No. 658, the Edible Fats (Standard of Quality) Order, 1919.



REVIEWS.

A DICTIONARY OF APPLIED CHEMISTRY. Vol. II.: Calculi to Explosion. By SIR EDWARD THORPE, C.B., F.R.S., assisted by eminent contributors. Revised and enlarged edition, 1921. London: Longmans, Green and Co. Price 60s. net.

A review of the first volume of this most comprehensive treatise has already appeared in the ANALYST (1921, p. 225), and the comments there made upon the general character of this edition apply equally to the second volume.

There is a further increase in the bulk; the subject matter of 547 pages of the 1912 edition now occupies 717 pages, to which must be added the space afforded by the deletion of a number of small articles, so that it will be apparent how much new matter has been incorporated. In addition to the bringing up to date of all the articles there are notable increases in the articles on the different dyestuffs and colouring matters, which, it is hoped, is a reflection of the gradual development of this industry in Britain.

A number of excellent articles on different alkaloids and drugs appear in this volume which were formerly grouped together in the general article on vegeto-alkaloids. For example, there is a new article of twenty-six pages by Howard and Chick, giving a very complete account of cinchona and its alkaloids. The articles on drugs reflect the considerable increase of knowledge of these substances during recent years. The reviewers would here venture a suggestion that the usefulness of some of these articles would be further enhanced by the inclusion of a brief account of the microscopic characteristics of the drugs for purposes of identification. It has been noticed, too, that there is an increased number of references to English chemical literature and abstracts; this is highly desirable in view of the time which may often be saved by reference to the English abstract of a foreign paper. Turning now to some of the articles in detail, a few points may be noted.

Under Carbohydrates there is a paragraph on heptoses, and mention is made of new sugars, and work done within the last few years is all incorporated. In view of the importance of cellulose esters in aeroplane manufacture the account of these substances included under cellulose might be somewhat enlarged, and a more complete list of references would be useful. The section on Cement does not appear to have been much altered from the 1912 edition, though a fuller account of the mechanical testing of cement is given. As the intermittent kiln is now practically obsolete, some space might be saved by the excision of the early types of these kilns. Under the heads of Chemical Affinity and Industrial Catalysis are found most interesting accounts of some of the newer industrial processes, including Haber's and Sabatier's reactions, and applications of enzyme reactions. The old article on Chromophores and Chromogens has disappeared and is replaced by a comprehensive

new article on Colour and Constitution, by Watson; this article is distinctly utilitarian; chemical theories are considered at some length, but physico-chemical theories are not discussed, because they have not led to the discovery of new dyes or the development of the industrial side of the subject.

The important subject of corrosion of metals and their protection therefrom is dealt with very fully. In addition to the articles on corrosion of iron and the fouling of ships, by Brame, there is a lengthy article on corrosion of metals in general by Friend. Although there are thus three separate articles on different aspects of the same general subject, there is little or no duplication of matter. The article on Colloids is comparatively short; it is surprising that no reference is given to the useful reports on this subject published under the auspices of the British Association.

A new article appears on Decolorising Carbons, in which is described in some detail the manufacture and special properties of different kinds of animal charcoal, which subject was deleted from Vol. I. The absorption of gases by charcoal is considered under the heading of Carbon.

The ingenious and useful film evaporator designed by Kestner now finds description in the revised article on Evaporators, and the article on Gaseous Explosion, rewritten by Bone and Wheeler, is quite up to date, and includes a summary of the large amount of new information worked out by Wheeler and his collaborators.

The new edition of this volume has, as a whole, been excellently revised, and there are few printer's errors; on p. 568, however, for "Dutton" should be "Dufton," and on p. 226 there should be inserted a reference to Wade and Finnemore (*Trans. Chem. Soc.*, 1904, 85, 938). There is so much new matter included that the technical chemist can scarcely afford to do without this edition of a unique treatise.

G. R. THOMPSON.

H. E. COX.

THE CHEMIST'S YEAR-BOOK, 1921. Edited by F. W. ATACK, M.Sc., B.Sc. Sixth Edition. Two vols., pp. 1123 and index. Manchester: Sherratt and Hughes, 1921. Price 21s.

This publication, previous editions of which have been reviewed in the ANALYST (1917, 42, 406), is now in its sixth edition, in two volumes. The book is to be regarded from several aspects. In the first place it is a book of chemical and physical data, and, as that, contains a variety of useful and important information. There are to be found here extended tables of specific gravities of solutions and of liquids, of solubilities of gases in liquids and of organic and inorganic solids, alcohol tables, tables of vapour pressures, constant boiling-point mixtures: gas volume corrections, tables, and many other data too varied to be enumerated. There are tables giving formula, density, solubility, and melting- and boiling-points of upwards of 1,000 inorganic and nearly 3,000 organic substances; others giving the composition and main physical properties of over 1,200 minerals, and the constitution of from 700 to 800 dyes. Another table gives the formulæ and certain properties of about 140 alkaloids. There are two very useful lists of the pharmaceutical names of synthetic compounds and of the trade names of drugs. In many cases the authority for the

practical worker and chemist in the mill who is desirous of possessing a general knowledge of the subject and its application.

Chapter II. describes the various fibres used in paper-making, with their chemical and microscopical characteristics; these are illustrated with an excellent set of photomicrographs prepared by the U.S. Paper Section of the Bureau of Standards, and should be found very useful to the student.

Chapters III. to VII. deal very fully with the preparation of the typical raw materials, the actual boiling trials, more especially in the treatment of wood by the soda, sulphate, and sulphite processes, being illustrated with various set examples of the composition of the liquors and methods of analysis. This detailed work is certainly a departure from the usual textbook method, and the author is to be congratulated on including analytical details.

Chapters VIII. to XI. deal with the incidental operations of bleaching, sizing, loading, and colouring of pulps.

One very important detail in a textbook of paper-making is lacking, and that is the operation of beating. It is usually claimed, and rightly so, that the finished paper is made in the beating room; hence the absence of any details of effects of beating upon the different fibres in the ordinary hollander, the various types of modern beaters with auxiliary circulation, and the incidental details of bed-plate and weight of beater rolls to hydration of pulp detracts somewhat from the value of the book to the paper-making student.

The same remarks may be applied to the absence of any details of paper-making machines—that is, Fourdrinier, mould and board machines. The chapters on coating and paper-testing follow the lines usually laid down in a book of this kind.

W. BACON.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS. 6 DEC. 1921

OBITUARY NOTICE.

EDWARD JOHN BEVAN.

EDWARD JOHN BEVAN was the son of Edward Bevan, of Birkenhead. He was born in 1856, and was educated at private schools in Birkenhead and Birmingham. As he had shown the boy-amateur's predilection for experimental "science," his parents secured for him, at the age of seventeen, a post as assistant in the laboratories of the Runcorn Soap and Alkali Company, and the opportunity of a career as chemist.

At this early stage he came under the influence of pioneer workers in the alkali industry, the Brothers Hargreaves, Davis, and Weldon, who wisely advised systematic academic training and the "genial" atmosphere of Owens College, at that date (1876) the recognised head centre of chemical science in this country.

Here he was associated with C. F. Cross and a group of research workers, under the influence of Roscoe and Schorlemmer, and the student friendship which resulted was so cemented that on leaving the college, each to engage on technical research work in connection with "cellulose" industries, active correspondence and in fact collaboration ensued. Bevan's position was that of chemist at the Musselburgh Paper Mill (A. Cowan and Co.), where he enjoyed the sympathetic collaboration of the director-manager, R. C. Menzies, which contributed much to the consolidation of his technical-scientific grasp of manufacturing industry. Further, his three years' residence in the bracing atmosphere of the East of Scotland changed Bevan in point of health from the "rather delicate" youth to a man of relatively robust constitution.

The collaboration with C. F. Cross then resulted in the momentous decision for a joint adventure in the field of cellulose research, pure and simple, and, coming to London, they were able, through the influence of such friends as Thiselton Dyer, Hugo Müller, and H. E. Armstrong, to secure an entrée to the Jodrell Research Laboratory, Kew Gardens (1883). The problem of "ways and means" asserting itself on the prospective exhaustion of the slender funds accumulated in the previous period of salaried appointments, it was necessary to supplement the research programme by work in the technical-industrial field. There resulted the more formal partnership of "Cross and Bevan," an engagement with the firm of Thomson, Bonar, and Co. for research work in developing the "Ekman" bisulphite pulping process (wood cellulose), and the finding of laboratory accommodation, which they owed to R. E. H. Goffin and the Council of the Westminster (Secondary) Schools.

This was the early phase of long years of struggle which has resulted in the achievements by which "Cross and Bevan" is or are known.

But it is due to Edward John Bevan, as a Past-President of the Society, to notice more particularly that phase of his career in which he has made his special personal reputation—that is, in filling the position of Public Analyst under appointment by the Middlesex County Council.

Apart from his official duties, he took, from the first, a keen interest in the work of our Society. He was an abstractor of the *ANALYST* in its early days, and was a frequent contributor to its pages. For ten years (1894–1904) he was one of the Secretaries, in which position his enthusiasm and unfailing courtesy endeared him to all the members. In the following year he was elected President.

His professional reputation is so well established, generally, and, as Bevan would say, generously recognised, that the writer can add nothing to the estimates of his achievement in this field, often and fully expressed by his brother chemists, who, from their training and the exacting discipline of their professional work, are of necessity rigorous in their judgment of capacity.

The writer knows that Bevan derived the greatest encouragement from this ungrudging recognition by his peers of his actual professional work and its associated activities for the general welfare, of which mention should also be made of his devoted work as Fellow, Member of Council, and Vice-President of the Institute of Chemistry.

What the writer feels competent to add is a word on the moral qualities which Bevan brought to bear on his work—a grasp of order and method necessarily; a finished skill in manipulation, with great rapidity in routine operations; and, above all, a sensitive conscientiousness in regard to accuracy and the rigorous discipline, self-imposed, in taking all possible trouble to exclude every error outside the inevitable but measurable error of all laboratory estimations.

Such qualities may no doubt, and in a measure, be acquired; in Bevan's case it was original endowment or genius.

That he was "genial," in the ordinary acceptance of the term, is especially known to his wide circle of intimate friends. His was an attractive personality; he had much charm of manner, which made him popular in many charmed circles of club and of family, and his fondness for outdoor sports of all kinds was an added link of interest with many friends both inside and outside his profession. In his youth he was a keen cyclist, and later actively welcomed the coming of the motor-car, whilst still more recently he became an ardent golfer. He was also particularly interested in theatrical matters, and was for many years a supporter of the Playgoers Club. He was not married.

Those who may be interested in the records of research work in which the names of C. F. Cross and E. J. Bevan are associated, are referred to an article in the *World's Paper Trade Review* for January 28, 1921, from which it will be seen that the association has weathered the struggles and storms of forty-two years.

As a last word, Bevan's death, on October 17, at the early age of sixty-five, was directly connected with his personal war service—the conscientious discharge of his duties as "special" in his home district (Watford).

C. F. CROSS.

NOTE.—A portrait of Mr. Bevan will be published in a forthcoming number.—EDITOR.

THE SEPARATION OF ALUMINIUM FROM BERYLLIUM.—PART II.*

By HUBERT T. S. BRITTON, B.Sc., A.I.C.

AMMONIUM CARBONATE METHOD.

THE separation of beryllium from aluminium depending on the insolubility of aluminium hydroxide and the solubility of beryllium hydroxide in concentrated solutions of ammonium carbonate was discovered by Vauquelin in 1798 (*Ann. Chim. Phys.*, 1798, 26, 155). Damour (*ibid.*, 1843, [iii.], 7, 173) regarded the method as unreliable, as he found that it gave too low results for beryllium. It was first described in detail by Rose (*Handbuch der Analyt. Chem.*, 1851, II., 59). Two procedures were given: treating either (a) the hydroxides, or (b) the solutions of the two metals, with a sufficiently large quantity of ammonium carbonate solution. He admits that a little aluminium is afterwards found in solution. Wöhler, however (*Handbook of Inorg. Analysis*, English edition, 1854, 114), states that aluminium hydroxide can be completely precipitated by slowly pouring a solution of the two earths into an excess of a warm concentrated solution of ammonium carbonate. Weeren (*Pogg. Ann. der Physik.*, 1854, 92, 91) found that the method yielded an incomplete separation, inasmuch as some alumina passed into the ammonium carbonate solution. Lewy (*Comptes rend.*, 1857, 45, 881) used the method for the analysis of beryl. Hofmeister (*J. prakt. Chem.*, 1859, 76, 1) endorsed Weeren's observation that the ammonium carbonate solution dissolved some aluminium hydroxide, and endeavoured to remove it by a series of fractional precipitations of the hydroxides contained in the filtrate. By such a treatment he obtained a slightly higher figure for alumina than that obtained by Lewy for the same type of beryl. Joy (*Sill. Amer. J. Sci.*, 1863 [ii.], 36, 83) agreed with previous investigators that considerable amounts of alumina were dissolved by ammonium carbonate solutions. He carried out his experiments by pouring the solution of aluminium and beryllium salts into a warm saturated solution of ammonium carbonate and allowing it to stand, with occasional stirring, for some days. He investigated the solubility of beryllium hydroxide in a saturated solution of ammonium carbonate when allowed to stand, with occasional shaking, in stoppered bottles for varying times, and found that the maximum solubility was reached after ten days, and, further, that the solubility then decreased by as much as 15 per cent. after sixteen days. Williams (*Proc. Roy. Soc.*, 1877, 26, 165) thoroughly investigated the method, and established the fact that beryllium hydroxide was "permanently soluble in a saturated solution of carbonate of ammonium," his solubility experiments extending over three years at room temperature. When the same solution (4 grms. BeO per litre) was heated in a sealed tube at 100° C. for two days, he observed that no precipitate formed. Williams also was not able to confirm the observations of Weeren, Hofmeister, and Joy that some alumina passed into solution, but, on the contrary, found that some beryllia was always precipitated with the alumina when allowed to stand for periods varying

* See ANALYST, 1921, p. 359.

from five minutes to forty-eight hours. In the separation, in which he tried to remove the beryllia from the first aluminium hydroxide precipitate, he found that no less than seven extractions were necessary to reduce it to as little as $\frac{1}{2}$ per cent. Wünder and Chéladzé (*Ann. Chim. anal.*, 1911, 16, 205) examined the method by treating the precipitated hydroxides with a 5 per cent. ammonium carbonate solution for periods ranging from fifteen minutes to fourteen hours, and in every case found that much beryllium hydroxide remained undissolved.

The literature on this subject is thus somewhat confusing. On account of the results of some preliminary experiments, it was thought that the method might be made satisfactory, especially if carried out by gradually pouring a solution of aluminium and beryllium salts into a *cold* concentrated solution of ammonium carbonate, with continuous stirring. In this way it was hoped to minimise as much as possible any adsorption effects, it being thought that the failure of Wünder and Chéladzé was due to some beryllium hydroxide being occluded by the aluminium hydroxide, and consequently not being acted on by the ammonium carbonate solution. The aluminium hydroxide which is precipitated by means of ammonium carbonate is flocculent, and is therefore easily filtered and washed.

EXPERIMENTAL.

The behaviour of ammonium carbonate solutions towards solutions of aluminium sulphate and beryllium sulphate was tested in the following manner: To 100 c.c. of saturated ammonium carbonate solution 0.5 *N*-beryllium sulphate solution was added gradually from a burette, with shaking, until a precipitate was just produced; 200 c.c. of the 0.5 *N*-beryllium sulphate solution were required—in other words, 100 c.c. of saturated ammonium carbonate solution, even when diluted to 300 c.c., are capable of maintaining 1.255 grms. of beryllium oxide in solution. The experiment was repeated with a boiling ammonium carbonate solution. Nothing happened until 60 c.c. of 0.5 *N*-beryllium sulphate solution had been added, when an intense precipitate was immediately produced, which slowly dissolved on the addition of another 100 c.c. of ammonium carbonate solution.

In the case of aluminium, a distinct precipitate was formed by the addition of 0.1 c.c. of 0.5 *N*-aluminium sulphate solution to 100 c.c. of ammonium carbonate solution at room temperature. With boiling ammonium carbonate, some alumina passed into solution, no precipitate being produced until 2.8 c.c. of 0.5 *N*-aluminium sulphate solution had been added (*i.e.*, = 0.024 gm. Al_2O_3).

Whereas the effect of heating the ammonium carbonate solution was to cause the solution of some aluminium hydroxide sufficient to render unreliable any separation involving the use of *warm* ammonium carbonate, separations in the *cold*, if no other complications arose, should be quantitative.

For the following separations, solutions were made up containing weighed amounts of beryllium sulphate and aluminium sulphate.

Experiment 1.—To 100 c.c. of a solution of ammonium carbonate a solution of beryllium sulphate was slowly added, with continuous shaking, the beryllium passing completely into solution; then a solution of aluminium sulphate was slowly added with shaking. The precipitate of aluminium hydroxide appeared from the

first. It was filtered immediately by suction, washed with ammonium carbonate solution, and ignited.

Experiment 2.—To a solution of beryllium sulphate and aluminium sulphate 100 c.c. of saturated ammonium carbonate solution were added. This caused a precipitate, which, on shaking, dissolved to almost a clear solution. After further shaking and vigorous stirring the solution became opalescent in appearance, and in about five minutes a heavy precipitate settled out. When no further precipitate appeared to separate out, the precipitate was filtered, washed, and estimated.

Experiment 3.—A solution of the two sulphates was slowly added, with stirring, to 100 c.c. of a saturated solution of ammonium carbonate. This caused no apparent change, but on stirring the solution gradually became opalescent, and a precipitate finally settled out. It was allowed to stand for ten minutes, filtered, washed, and ignited.

Experiment 4.—As it was believed that the delayed formation of the colloidal precipitate observed in Experiments 2 and 3 was probably the cause of the beryllia being found in the aluminium hydroxide precipitate, this experiment was carried out as follows: To a solution of aluminium and beryllium salts 100 c.c. of the saturated ammonium carbonate solution were added drop by drop, with stirring, in order that the two hydroxides might first be precipitated in extremely small amounts, and that the addition of more ammonium carbonate might dissolve the beryllium hydroxide. After the ammonium carbonate was all added, the solution was vigorously shaken for five minutes, and then filtered by suction. The filtrate at first was clear, but after a time it became opalescent, finally gave a precipitate, and was filtered. Both precipitates were washed with ammonium carbonate solution and estimated. First precipitate = 0.0997 grm.; second precipitate = 0.0547 grm.

Experiment Number.	Grm. Taken.		Grm. Al_2O_3 Found.	Extra Grm.
	BeO.	Al_2O_3 .		
1	0.1278	0.1744	0.2224	0.0480
2	0.1805	0.2406	0.3070	0.0664
3	0.1579	0.2117	0.3020	0.0903
4	0.1261	0.1278	0.1544	0.0266

From these results it will be seen that in every case the amount of beryllia carried down by the precipitated aluminium hydroxide was appreciable, no matter how the process was carried out. Nor could the beryllia be removed from the alumina precipitate, although as much as 200 c.c. of saturated ammonium carbonate solution were employed in washing. There seems no doubt that the delayed precipitation was the cause of the failure to effect a quantitative separation. Experiments were tried, having either the sulphate or chloride of potassium present in solution, in the hope that such an electrolyte would promote immediate precipitation of the aluminium hydroxide, but the results were negative.

It was now left to be decided whether ammonium carbonate might be used under the conditions of the previous experiments as a means of obtaining beryllium hydroxide free from alumina. It seemed reasonable to regard the aluminium hydroxide precipitate as containing all the alumina, together with the beryllia which it had carried down, and thereby leaving only beryllia in solution. As the presence of aluminium hydroxide renders some beryllia insoluble, it was thought just possible that the converse, beryllia rendering some alumina soluble, might also be true. Two separations were therefore carried out in the manner described in Experiment 4. The filtrates were evaporated to dryness, during the course of which the beryllium hydroxide was precipitated. The residues, which had not been heated too strongly, were dissolved in dilute hydrochloric acid, and heated to drive off carbon dioxide; then excess of about 6 *N*-sodium hydroxide solution was added, and the solution again boiled to drive off ammonia. The solutions, having been evaporated to about 20 c.c., were cooled, and sufficient hydrochloric acid added to produce the faintest precipitates, the hydroxides thus being dissolved in the minimum amounts of sodium hydroxide solution. Each of the solutions was then diluted to 500 c.c., boiled for forty minutes, and filtered. Under these conditions no beryllia should remain in the mother liquor. On acidification with hydrochloric acid and treatment with ammonium chloride and ammonium hydroxide a gelatinous precipitate was produced in both filtrates. These precipitates were proved to be aluminium hydroxide by dissolving one in a little potassium hydroxide solution, and, after having driven off any occluded ammonia by boiling, acidifying the solution with sulphuric acid and leaving it to crystallise; and the other, by suspending it in a saturated solution of ammonium chloride and boiling the liquid for several hours until all free ammonia had been driven off. In the first case crystals of alum were obtained, and in the second the precipitate did not dissolve. Hence, all alumina is not precipitated by the addition of ammonium carbonate in the cold when beryllium salts are present in solution.

In conclusion, the ammonium carbonate method is unsatisfactory both quantitatively and qualitatively.

AMMONIUM SULPHITE METHOD.

Two methods of separation of aluminium from beryllium were described by Berthier (*Ann. Chim. Phys.*, 1843, [iii.], 7, 74), which depend upon the fact that the concentration of the hydrogen ions in a weak solution of sulphurous acid from which no more sulphur dioxide can be expelled by boiling is sufficient to maintain beryllium hydroxide in solution, but insufficient to prevent the precipitation of aluminium hydroxide. One method was to dissolve the hydroxides of aluminium and beryllium in sulphurous acid, and then boil until the evolution of sulphur dioxide had ceased; whilst the other was to treat a solution of the two hydroxides with excess of a strong solution of ammonium sulphite, and boil until all sulphur dioxide had been driven off. Böttiger (*Liebig Ann.*, 1844, 51, 397) investigated these methods, and found that in both cases some beryllia was invariably precipitated with the alumina. Weeren (*Pogg. Ann. der Physik.*, 1854, 92, 91) and Joy (*Sill. Amer. J. Sci.*, 1863, [ii.], 36, 88) arrived at the same conclusion.

The two methods are in reality almost identical, for in the case where the

precipitated hydroxides are employed much ammonia is retained in the precipitates. It was therefore decided, first of all, to ascertain under what conditions the aluminium hydroxide was completely precipitated and the beryllium hydroxide completely soluble.

EXPERIMENTAL.

A solution of ammonia (sp. gr. 0·880) was saturated with sulphur dioxide. On adding 0·5 *N*-beryllium sulphate solution in considerable quantity to 100 c.c. of the boiling solution no precipitation occurred, even after boiling for several hours until all sulphur dioxide had been given off. The solution remained clear, even when evaporated to the point of crystallisation. When 0·5 *N*-aluminium sulphate solution was added to 100 c.c. of the boiling sulphite solution, no precipitation took place until 0·5 c.c. had been added, when a distinct turbidity was produced, more aluminium sulphate producing a precipitate. After boiling a solution containing much suspended aluminium hydroxide until the evolution of sulphur dioxide had ceased, no aluminium hydroxide could be found in the filtrate. The aluminium hydroxide precipitate was not gelatinous, and could be filtered easily. As the fundamental principles of this process were satisfactory, two separations were carried out thus : Solutions containing weighed quantities of the two sulphates were each added to 100 c.c. of ammonium sulphite solution. In each case no precipitation occurred at first, but after a few seconds the solutions gradually became cloudy, and precipitates ultimately settled out. Boiling was carried on, keeping the solutions up to their original volume by occasionally adding water until no sulphur dioxide could be detected in the issuing steam, after which the precipitates were filtered off, ignited, and weighed.

Number.	Grm. Taken.		Grm. Al_2O_3 Found.	Extra Grm.
	BeO.	Al_2O_3 .		
1	0·1081	0·1695	0·1811	0·0116
2	0·0706	0·0766	0·0911	0·0145

As the weights of alumina in both cases were much too high, due to the precipitation of the beryllium hydroxide under the influence of the aluminium hydroxide, the mother liquors were examined for the presence of alumina in a similar way to that described in the previous section, but none was found.

Further analyses were considered unnecessary, as the method appeared to give analogous results with the ammonium carbonate one, due in all probability to the same cause—viz., the delayed formation of the aluminium hydroxide precipitate. Although it has not been possible to render this separation quantitative, it can be employed for freeing beryllium hydroxide from alumina. The latter procedure, however, involves much loss of beryllia and is tedious, many hours' boiling being required to drive off all the free sulphur dioxide.

SODIUM CARBONATE AND SODIUM BICARBONATE METHODS.

Hart (*J. Amer. Chem. Soc.*, 1895, 17, 604) stated that adding sodium carbonate to a solution of aluminium and beryllium salts caused the precipitation of nearly all the alumina, the beryllia remaining in solution. The suggestion was improved upon by Parsons and Barnes (*J. Amer. Chem. Soc.*, 1906, 28, 1589), who recommended the use of a 10 per cent. solution of sodium bicarbonate, rapidly raising the temperature of the solution to boiling, and keeping it at that temperature for not more than half a minute. Their method was to dissolve convenient quantities of the hydroxides in hydrochloric acid, neutralise as nearly as possible with ammonium hydroxide, make up to 100 c.c., and then add 10 grms. of sodium bicarbonate. The mixture was heated rapidly to boiling, and kept boiling for half a minute, cooled, and filtered. The aluminium hydroxide precipitate contained some beryllia, which, according to Parsons and Barnes, can be removed by another re-solution and re-extraction. Noyes, Bray, and Spear (*J. Amer. Chem. Soc.*, 1908, 30, 481) agree that the method is satisfactory when the amount of alumina present is not large. When the alumina present was between 0.1 and 0.5 gm., they found that the aluminium hydroxide precipitate retained from 0.002 to 0.005 gm. of beryllium oxide. As the author did not obtain good separations by this method in several instances, it was decided to investigate it in order to find what conditions were necessary to make it give accurate results.

EXPERIMENTAL.

The following Table I. gives the number of c.c. of 0.5 *N*-beryllium sulphate solution and 0.5 *N*-aluminium sulphate solution which can in each case be added to 100 c.c. of sodium carbonate solutions before a precipitate is formed:

TABLE I.

	Temperature.	Sodium Carbonate saturated at 15° C.	0.502 <i>N</i> -Na ₂ CO ₃ .
0.5 <i>N</i> -BeSO ₄ ... {	15° C.	150 c.c. = 0.9412 gm. BeO.	0.1 c.c. = 0.0006 gm. BeO.
	100° C.	150 c.c. = 0.9412 gm. BeO.	0.25 c.c. = 0.0015 gm. BeO.
0.5 <i>N</i> -Al ₂ (SO ₄) ₃ {	15° C.	7.0 c.c. = 0.0596 gm. Al ₂ O ₃ .	5.3 c.c. = 0.0451 gm. Al ₂ O ₃ .
	100° C.	6.5 c.c. = 0.0554 gm. Al ₂ O ₃ .	5.0 c.c. = 0.0426 gm. Al ₂ O ₃ .

From these data it will be seen that the solubility of aluminium hydroxide in a saturated solution of sodium carbonate is sufficient to render a method depending upon it futile. The results obtained from 0.502 *N*-sodium carbonate solution show that the effect of diluting sodium carbonate solutions of alumina and beryllia is to precipitate both the hydroxides:

Table II. gives similar results for sodium bicarbonate solutions.

TABLE II.

	Tempera- ture.	Sodium Bicarbonate.			
		0·942 <i>N</i> · (Saturated at Room Temp.).	0·687 <i>N</i> ·	0·502 <i>N</i> ·	0·251 <i>N</i> ·
0·5 <i>N</i> -BeSO ₄ . c.c. = grms. BeO	15° C. {	23·2 0·1456	16·5 0·1035	10·0 0·0628	2·7 0·0017
0·5 <i>N</i> -BeSO ₄ . c.c. = grms. BeO	100° C. {	35·4 0·2221	22·0 0·1381	14·5 0·0910	2·7 0·0017
0·5 <i>N</i> -Al ₂ (SO ₄) ₃ . c.c. = grms. Al ₂ O ₃	15° C. {	0·05 0·0004	0·05 0·0004	0·05 0·0004	0·05 0·0004
0·5 <i>N</i> -Al ₂ (SO ₄) ₃ . c.c. = grms. Al ₂ O ₃	100° C. {	0·05 0·0004	0·05 0·0004	0·05 0·0004	0·05 0·0004

In the case of experiments carried out at 100° C., it should be stated that some beryllium hydroxide separated out on cooling. Table II. shows that Parsons and Barnes's method is theoretically possible when the amounts of beryllia present are within its limits of solubility in the amount and concentration of the sodium bicarbonate solution employed. If the separation is carried out by taking a neutral solution of about 20 c.c. of the two earths, to which 100 c.c. of saturated sodium bicarbonate solution are to be added, it appears that the beryllia should not exceed 0·15 gm. When the neutral solution is made up to 100 c.c., and 10 grms. of sodium bicarbonate are added, a little more than 0·15 gm. of beryllium oxide may be present. However, as a safe working rule, the amount of beryllia present should be less than 0·15 gm.

Experiments were carried out to ascertain what was the effect of boiling saturated sodium bicarbonate solutions in which (a) aluminium hydroxide was suspended, and (b) beryllium hydroxide was dissolved. After boiling for ten minutes, not a trace of alumina could be found in the mother liquor, but in the case of the beryllium hydroxide solution a slight precipitate began to deposit. From Table I. it will be observed that alumina is slightly soluble in boiling sodium carbonate solutions; hence it is possible that the decomposition produced by prolonged boiling of a bicarbonate solution may cause the solution of a little alumina. Solution of beryllium hydroxide in sodium bicarbonate solutions takes place much more quickly on warming; consequently, as Parsons and Barnes suggested, it is advisable to carry out the separation by rapidly raising the temperature to boiling.

The following separations were carried out with a view to finding the amount of beryllia which is carried down with the aluminium hydroxide in a single precipitation. Solutions were made up of weighed amounts of beryllium sulphate and aluminium sulphate in the minimum amount of water, and treated with saturated sodium bicarbonate solutions in various ways. The resulting mixtures were immediately filtered by suction, and the alumina estimated, after it had been freed from adsorbed

sodium salts, either by solution in hydrochloric acid and reprecipitation with ammonium chloride and ammonium hydroxide, or by extracting the ignited precipitate (having been ignited in a platinum crucible) with hydrochloric acid, decomposing any aluminate with ammonium chloride and ammonium hydroxide, and boiling the liquid.

TABLE III.

No.	BeO Taken.	Al ₂ O ₃ Taken.	Al ₂ O ₃ Found.	Amount BeO Adsorbed.	BeO in Alumina Precipitate.
	Grm.	Grm.	Grm.	Grm.	Per Cent.
1	0.1007	0.0262	0.0284	0.0022	7.7
2	0.0124	0.1176	0.1250	0.0075	6.0
3	0.0929	0.1644	0.1730	0.0086	5.0
4	0.1003	0.0259	0.0279	0.0020	7.2
5	0.1010	0.0512	0.0575	0.0063	11.0
6	0.1004	0.1020	0.1160	0.0140	12.0
7	0.0125	0.1175	0.1260	0.0085	6.7
8	0.0560	0.1025	0.1045	0.0020	1.9
9	0.1103	0.0794	0.0837	0.0043	5.1
10	0.0132	0.1087	0.1090	0.0003	0.3

All these separations, except Nos. 1 and 2, having been mixed in the cold, were carried out by pouring a solution of the two sulphates drop by drop into 100 c.c. of warm, saturated sodium bicarbonate solution, and then quickly raising the temperature to boiling, keeping it at that point for not more than a minute, filtering the liquid, and making the estimation. In each case the aluminium hydroxide precipitation occurred from the beginning. It will be seen that the amount of beryllium hydroxide carried down with the aluminium hydroxide depended on (a) the bulk of the aluminium hydroxide precipitate, and (b) the amount of beryllia in solution. It was thought that the beryllium hydroxide which was brought down was mechanically adsorbed by the aluminium hydroxide, and consequently vigorous stirring should have the effect of forcing much of the adsorbed beryllia back into solution. This was borne out in separation No. 8, where, the mixture having been kept thoroughly stirred, the amount of beryllium oxide adsorbed was only 1.9 per cent. Stirring in the case of No. 9 did not have as great an effect, as the amount of beryllia present was proportionately greater. As the ratios of the two oxides taken in separations Nos. 1 to 9 are quite general, it was inferred that with efficient stirring the amount of adsorbed beryllia under the same conditions was never likely to be more than 10 per cent. of the total precipitated oxides. The success of the method, involving a second precipitation from a neutral solution of the first precipitate, therefore depended upon whether a mixture of the two oxides, 10 per cent. of which was beryllia, could be quantitatively separated. Such a mixture was satisfactorily separated—viz., No. 10—the amount of beryllia being actually 10.8 per cent.

The following separations were carried out, employing two precipitations, by dissolving the first aluminium hydroxide precipitate in the minimum amount of

dilute hydrochloric acid, neutralising the solution with ammonia, and adding it drop by drop, with stirring, to 100 c.c. of warm saturated sodium bicarbonate solution, rapidly heating the liquid to boiling-point and allowing it to boil for half a minute, and completing the estimation of alumina as previously described. The beryllia was estimated as usual, after having combined the two filtrates and boiled the liquid with excess of concentrated hydrochloric acid until all carbon dioxide had been driven off.

TABLE IV.

No.	Grm. Al_2O_3 .		Grm. BeO .	
	Taken.	Found.	Taken.	Found.
1	0.0226	0.0226	0.1409	0.1406
2	0.0792	0.0795	0.1106	0.1106
3	0.0852	0.0855	0.0941	0.0940
4	0.1537	0.1542	0.1421	0.1417
5*	0.1087	0.1090	0.0132	0.0129

If the necessary precautions are taken, the method is therefore capable of giving satisfactory results.

SUMMARY.

(1) Ammonium carbonate is of no use for the quantitative analysis of solutions of aluminium and beryllium salts; some beryllia is carried down with the alumina, and also a little alumina remains in solution.

(2) Ammonium sulphite does not give a quantitative separation, as some beryllia is absorbed by the alumina. All the alumina is, however, precipitated.

(3) A saturated solution of sodium carbonate dissolves an appreciable quantity of beryllia, but also much alumina.

(4) The method employing two precipitations by means of saturated sodium bicarbonate solution is satisfactory, provided that (a) neither the beryllia nor the alumina in 100 c.c. of solution saturated with sodium bicarbonate is more than 0.15 gm.; (b) adsorption in both precipitations is kept at a minimum by thorough stirring.

The author desires to take this further opportunity to express his appreciation of the interest taken by Professor A. J. Allmand in this work.

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W.C. 2.

* These data give the complete analysis of mixture No. 10 in Table III., and were therefore obtained by one precipitation only.

SUGAR CALCULATIONS.

By J. F. LIVERSEEGE, F.I.C.

A PUBLIC ANALYST in the examination of substances containing sugars has to use constants and equations. The following notes are the results of an attempt, by selection and calculation, to obtain a set of data that is clear and consistent.

SPECIFIC ROTATORY POWER.—There are ambiguities in published specific rotatory powers owing to authors failing to state the temperature and concentration of the solutions used, and sometimes it is not clear whether the sugar was anhydrous or contained water of crystallisation. In some cases results given by various authorities differ considerably, and the selection of the best value has been difficult. This particularly applies to *lævulose* and invert sugar. The value for invert sugar should be half the algebraic sum of those of *lævulose* and *dextrose*. The selected values for sugars are given in the table on p. 450. In practice it is a convenience to apply the term "specific rotatory power" not only to pure sugars, but also to mixed substances such as treacle.

CORRECTION FOR TEMPERATURE.—In factors given for the correction of specific rotatory power there often is doubt as to whether in the equations given the sign for left-handed rotation (–) is to be used as well as any algebraical sign. As the temperature correction will never alter the rotation from "+" to "–," it is better to ignore the optical signs in making this correction.

Equation (1), given below, is to be used when the specific rotatory power of a pure sugar is being determined; equation (2) when the actual angle of determination is being corrected for temperature. In this case, α^{20} means the angle at 20° C. of a solution examined in a 200 mm. tube; $[\alpha]_D$ may be substituted for α in this equation. The values for " D " and " d " are given in the table, and " t " represents degrees C.

$$(1) [\alpha]_D^{20} = [\alpha]_D^t + D(t - 20). \quad (2) \alpha^{20} = \frac{\alpha^t}{1.0 + d(20 - t)}.$$

The application of equation (2) may be illustrated by the correction for temperature of a solution of invert sugar, which gave a rotation of -7.7° at 24.4° C.:

$$\alpha^{20} = \frac{7.7}{1.0 + 0.0149(20 - 24.4)} = 8.24^\circ.$$

Solutions should be made up to volume at 20° C., and polarised as near this standard temperature as is convenient.

CORRECTION FOR CONCENTRATION.—The standard concentration is 10 *w/v* (*i.e.*, 10 grms. per 100 c.c.). When a sugar solution is diluted, in addition to the primary reduction of the angle according to the equation,

$$(3) \alpha = \frac{w/v \times [\alpha]_D}{50},$$

there is with some sugars a secondary reduction, owing to the fact that in dilute solutions the rotatory power *per unit of sugar* is less than in stronger solutions.

The sign $[\alpha]_D^{20}$ is often used to indicate the specific rotatory power of a sugar at

20° C. when a sodium flame is used. A useful extension of it is to add the figure for the w/v of the solution to the left-hand side of the bracket. The sign $^{10}[a]^{20}$ would therefore mean that the specific rotatory power was corrected to 20° C., and that the figure was also corrected for this secondary alteration to 10 w/v . The corresponding sign for the angle actually observed in a 200 mm. tube is $^{10}\alpha^{20}$. Again, to avoid ambiguities, the optical signs of the sugar solutions are ignored in making the correction.

Equation (4) is to be used for correcting the specific rotatory power of sugar to the standard of 10 w/v when the concentration is known. In many cases, however, the concentration of the solution has to be determined, and therefore cannot be used. In such cases an approximation may be made by using the determined angle in place of the w/v , as in equation (5). The values for "C," "c," and "B," are taken from the table :

$$(4) \quad ^{10}[a]_D = ^r[a]_D + C (10 - w/v).$$

$$(5) \quad ^{10}\alpha^{20} = ^r\alpha^{20} + c (B - \alpha^{20})^r\alpha^{20}.$$

The above-mentioned solution of invert sugar may be taken as an example of equation (5) :

$$^{10}\alpha = 8.24 + 0.0080 (4.2 - 8.24) 8.24 = 7.98^\circ.$$

CALCULATION OF w/v WHEN ONE SUGAR ONLY IS PRESENT.—Either of the following equations may be used :

$$(6) \text{ Sugar } w/v \text{ in the solution} = \frac{50 \times ^{10}\alpha^{20}}{^{10}[a]_D^{20} \text{ of pure sugar}}.$$

$$(7) \text{ Sugar } w/v \text{ in the solution} = \frac{10 \times ^{10}\alpha^{20}}{B}.$$

Applying this to the above-mentioned solution of invert sugar,

$$\frac{50 \times 7.98}{21} \text{ or } \frac{10 \times 7.98}{4.2} = 19.0 \text{ } w/v;$$

and as 100 c.c. contained 19.2 grms. of the substance, the percentage of invert sugar present = $\frac{19.0 \times 100}{19.2} = 99$ per cent.

With specific rotatory powers the following equation may be used :

$$(8) \text{ Sugar per cent. (by weight) in the substance} = \frac{100 (^{10}[a]_D^{20} \text{ of substance})}{^{10}[a]_D^{20} \text{ of pure sugar}}.$$

MIXTURE OF TWO SUGARS.—Solutions of sucrose which is partly inverted often require examination. On inversion, 100 parts of sucrose yield 105 parts of invert sugar. In many cases it is more convenient to express invert sugar in terms of the sucrose from which it was derived, and I have used the term "inverted sucrose" to indicate this result. Unless it is known that only two sugars are present, it is necessary to prove by the determination of the reduction and total organic solids that such is the case. A mixture of sucrose and invert sugar in suitable proportions is optically inactive.

SUCROSE AND INVERTED SUCROSE.—When the substance consists of sucrose which has been partly inverted, the following equations will give its composition. "I" is used for the angle after inversion, corrected for temperature and concentra-

tion by equations (2) and (5), and α for the original angle of polarisation at the same dilution as " I ":

- (9) Sucrose w/v in solution = 0.565 (change of angle of rotation).
- (10) Inverted sucrose w/v originally present = 0.565 ($3.023 I \pm \alpha$).
- (11) Angle due to sucrose = 0.75 (change of angle of rotation).
- (12) Angle due to inverted sucrose originally present = 0.249 ($3.023 I \pm \alpha$).

In equations (9) and (11) the change of rotation will be the sum of the angles if the sign of rotation is altered by inversion, and their difference if their signs are the same.

In equations (10) and (12) " $-$ " is used if the sign of the optical rotation is changed by inversion. If these equations give a negative result, dextrose, and not inverted sucrose, is present.

SUCROSE AND DEXTROSE.—The corresponding equations for mixtures of sucrose and dextrose are:

- (13) Dextrose w/v = 0.235 ($\alpha \pm 3.023 I$).
- (14) Angle due to dextrose = 0.249 ($\alpha \pm 3.023 I$).

The " $-$ " sign is used if inversion changes the optical sign.

DEXTROSE AND LÆVULOSE: INVERT SUGAR.—If dextrose and lævulose are present in equal quantities they become invert sugar. If the quantities are dissimilar, a mixture of invert sugar with excess of lævulose or dextrose results, and the composition can be calculated in either form by the following equations:

- (15) Per cent. of invert sugar = 1.35 ($52.8 - {}^{10}[\alpha]^{20}$).
- (16) Per cent. of lævulose = 0.678 ($52.8 - {}^{10}[\alpha]^{20}$).

In these equations the optical signs must be included as well as the algebraic ones.

CORRECTION FOR TEMPERATURE AND CONCENTRATION OF A MIXTURE OF TWO SUGARS.—As sucrose and inverted sucrose polarise in opposite directions, the angle of a mixture of them is not a measure of the amount of inverted sucrose that is present. To correct the original angle, it is necessary to make an approximate calculation of the amount of inverted sucrose present in the solution before inversion.

The figures obtained with a sample of syrup of rhubarb may be taken as an example of this correction. A 25.5 w/v solution of it gave ${}^{\alpha}{}_{16} = +6.3^{\circ}$ and ${}^{\alpha}{}_{12} = -7.0^{\circ}$. The latter, corrected by equations (2) and (5), became ${}^{10}I^{20} = -6.16$.

The approximate angle of the original inverted sucrose calculated by equation (12) = 0.249 ($3.023 \times 6.16 - 6.63$) = 3.1° .

This angle (-3.1) is therefore a measure of the inverted sucrose originally present, and can be corrected as usual by equations (2) and (5), which give the value -2.95° . The original angle can then be corrected by the result of this calculation, as the sucrose angle would not be affected by temperature and concentration:

$${}^{10}\alpha^{20} = 6.3 + (3.1 - 2.95) = 6.45^{\circ}.$$

The composition of the original solution can then be calculated by equations (9) and (10):

$$\text{Sucrose} = 0.565 (6.45 + 6.16) = 7.14 \text{ } w/v.$$

$$\text{Original inverted sucrose} = 0.565 (3.023 \times 6.16 - 6.45) = 6.88 \text{ } w/v.$$

As these were 25.5 *w/v* solutions, the syrup contained 28.0 per cent. of sucrose and 26.7 per cent. of inverted sucrose. The latter could be changed to invert sugar by multiplication by 1.05.

REDUCTION.—The following general equation for the calculation of *K*—i.e., reduction expressed as dextrose and lævulose—is sometimes useful :

If “*a*” grm. of sugar or syrup is diluted to “*b*” c.c. = (1),
and “*c*” c.c. of (1) is diluted to “*d*” c.c. by inversion = (2),
and “*e*” c.c. of (2) is diluted to “*f*” c.c. = (3),
and “*g*” c.c. of (3) = 10 c.c. of Fehling solution.

$$(17) K = \frac{5 b d f}{a c e g}$$

MIXTURES OF SEVERAL SUGARS.—When, in addition to the presence of sucrose, which is more or less inverted, glucose syrup is present, it is necessary to supplement the polarisation figures by a determination of the *K* and the organic solids both before and after fermentation. In such cases it is better to polarise at 20° C. instead of making the complicated temperature correction.

The fermentation may conveniently be performed as follows : Twenty grms. of the substance are weighed and mixed with 220 c.c. of water and 1 grm. of yeast, and the mixture is sterilised by boiling for a short time. After cooling, 3 grms. of yeast are added, and the flask is plugged with cotton-wool and incubated at about 28° C. for three or four days ; 100 c.c. of almost clear liquid are then decanted, and the remainder measured (= “*a*” c.c.). The 100 c.c. are evaporated down to less than one-half, alumina cream added, and diluted to $\frac{10,000}{100+a}$ c.c., so as to give a 20 *w/v* solution. After filtration the solution is analysed. It is a convenience to start two fermentations at once, so that if the *K* of the first indicated that fermentation is incomplete, the second flask can be incubated for a day or two longer, or the turbid liquid residue of the first fermentation may be returned to the incubator.

The following analysis of a sample of war-time “high-class stoneless damson and apple jam” shows the results obtained with such a mixture. The specific rotatory power before inversion was +47.3°, and after inversion +29.3°. These values, calculated by equations (11) and (8), give the specific rotatory power due to sucrose as 13.5°, and showed that 20.3 per cent. of sucrose was present.

					Organic Solids.	<i>K</i> .	[α] _D .
					Per Cent.		
Original	...	...	...	...	59.3	26	47.3
Subtracting sucrose	...	...	...	...	20.3	—	13.5
					39.0	26	33.8
After fermentation	...	...	...	...	16.0	3	24.6
Lost by fermentation	...	...	...	...	23.0	23	9.2

As the *K* of the fermented portion equalled the organic solids, the latter consisted of dextrose and lævulose. For this fermented sugar $[\alpha]_D = \frac{9.2 \times 100}{23} = 40.4^\circ$.

The percentage composition is then calculated by equation (15) :

$$\text{Invert sugar} = 1.85 (52.8 - 40.4) = 16.8 \text{ per cent.}$$

This shows that 16.8 per cent. of the 23 per cent. (= 3.9 per cent.) of the sugar fermented was invert sugar. The remainder of the *K* (26 - 3.9) may be taken to be dextrose. The optically active part of the 16.0 per cent. of residue after fermentation may be taken to be a mixture of dextrose and dextrin. The specific rotatory power due to the 3 per cent. of dextrose is subtracted.

				Organic Solids.	[α] _D .
				Per Cent.	
Unfermented	...	...	...	16	24.6
Dextrose	...	...	...	3	1.6
Dextrin, etc.	...	...	...	13	23.0

$$\text{Dextrin [by equation (8)]} = \frac{100 \times 23}{200} = 11.5 \text{ per cent. This leaves } (13.0 - 11.5 =)$$

1.5 per cent. of other unfermented solids.

The complete analysis of the jam was as follows : Sucrose, 20.3 ; invert sugar, 3.9 ; dextrose, 22.1 ; dextrin, 11.5 ; other soluble organic solids, 1.5 ; ash, 0.8 ; insoluble matter, 1.2 ; and water (by difference), 38.7 = 100.0.

A careful examination of the insoluble matter of the jam only detected 0.04 per cent. of damson skin ; so, in addition to being adulterated with glucose syrup, very little damson was present. There was some evidence that the jam had been dyed.

CONSTANTS AND FACTORS FOR SUGARS.

Sugar.	Molecular Weight.	$10[\alpha]_D^{20}$.	<i>B</i> .	<i>C</i> .	<i>c</i> .	<i>D</i> .	<i>d</i> .
Lactose :							
$C_{12}H_{22}O_{11}$...	342.24	55.3°	11.06°	—	—	0.079	0.0015
$C_{12}H_{22}O_{11} \cdot H_2O$...	360.26	52.5°	10.5°	—	—	0.075	0.0014
Maltose :							
$C_{12}H_{22}O_{11}$...	342.24	138°	27.6°	—	—	0.095	0.0007
$C_{12}H_{22}O_{11} \cdot H_2O$...	360.26	131°	26.2°	—	—	0.090	0.0007
Sucrose :							
$C_{12}H_{22}O_{11}$...	342.24	66.5°	13.3°	—	—	0.010	0.0001
Dextrose :							
$C_6H_{12}O_6$...	180.13	52.8°	10.56°	0.030	0.0005	—	—
Lævulose* :							
$C_6H_{12}O_6$...	180.13	95°	19.0°	0.110	0.0006	0.630	0.0066
Invert sugar* :							
$2(C_6H_{12}O_6)$...	360.26	21°	4.2°	0.070	0.0080	0.312	0.0149
Sucrose (inverted)* :							
$C_{12}H_{22}O_{11}$...	342.24	22°	4.4°	0.074	0.0077	0.327	0.0149
Dextrin :							
$n(C_6H_{10}O_5)$...	—	200°	40.0°	—	—	—	—

$^{10}[\alpha]_D^{20}$ = specific rotatory power of 10 w/v solution at 20° C. with sodium flame; B = rotation of 200 mm. 10 w/v solution at 20° C.; C = increase in numerical value of $^{10}[\alpha]_D^{20}$ for increase of 1 w/v of sugar; c = concentration coefficient; D = decrease in numerical value of $^{10}[\alpha]_D^{20}$ for increase of 1° C.; d = temperature coefficient; * = left-hand rotation.



NOTE.

The Editor desires to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

APPROXIMATE ESTIMATION OF COMMERCIAL CRESOL IN LYSOL.

THE process described in an abstract in the ANALYST (1921, 375) can be accurate only where the soap used is free from volatile fatty acids. Serious mistakes have been made by allowing volatile fatty acids (such as occur in coconut oil, etc.) to distil over and augment the layer of tar acids in the distillate. To obtain accurate results when there is any doubt of the quality of the soap, the following method may be employed:

Dissolve the sample in hot water in a separator, add a piece of stick sodium hydroxide, and shake the liquid until the alkali has dissolved. Add excess of brine and separate the precipitate. Redissolve the residue in hot water, add a small piece of stick sodium hydroxide, and precipitate the soap again with brine; repeat the process once more to make sure of extracting all the tar acids. Acidify the united alkaline solutions and extract them with benzene. The benzene can then be extracted with a small quantity of strong sodium hydroxide solution, and the latter can be acidified in a burette and the tar acids read off in the usual way.

G. F. WESLEY MARTIN.



NOTES FROM THE REPORTS OF PUBLIC ANALYSTS.

The Editor would be glad to receive the Annual or other Reports of Public Analysts containing matter of interest to the Society. Notes made from such Reports would be submitted to the Publication Committee.

ANNUAL REPORT OF THE BIRMINGHAM CITY ANALYST, 1920.

EGG POWDER.—Four of twelve informal samples were composed of tartaric acid, sodium bicarbonate, and rice starch. The other eight samples contained calcium hydrogen phosphate instead of tartaric acid, seven contained rice starch, and one a mixture of starches. One sample had been made with impure calcium hydrogen phosphate containing 55 per cent. of calcium sulphate, which is much in excess of the limit of 10 per cent. recommended for calcium hydrogen phosphate in a Local Government Board Report. The result was that the egg powder contained about 9 per cent. of calcium sulphate in excess.

The labels of some of the samples were very misleading, such as, for instance, "a perfect substitute for eggs." No action can be taken, however, for such misleading labels under the Food and Drugs Acts, although possibly the vendors may

be liable under the Merchandise Marks Act. The prices charged for the powders varied from 1d. to 2d. per ounce.*

CUSTARD POWDER.—Sixteen of nineteen informal samples consisted of coloured and flavoured starch, the starches found being maize, arrowroot, potato, tapioca, and rice. One sample, stated to contain 24 per cent. of proteid nourishment, or forty times as much as ordinary custard powder, contained only 5·4 per cent. of proteins. Another sample labelled "Sweetened with pure sugar only" contained about 50 per cent. of sugar; but two samples of the same make, marked "Sweetened, no sugar required," contained no sugar, but about 0·2 per cent. of saccharin. These two samples were marked as adulterated, and particulars of the case have been sent to the Ministry of Health.

REPORT FOR THE FIRST QUARTER, 1921.

NATIONAL HEALTH INSURANCE DISPENSING—Quinine Mixture.—An informal sample contained 21 grains of quinine sulphate and 183 minims of dilute hydrobromic acid, whereas the prescription ordered 32 grains and 240 minims respectively. A subsequent formal sample from the same vendor contained 19 grains of quinine sulphate and 206 minims of diluted hydrobromic acid. The explanation given was that the correct quantities had been dissolved in a Winchester quart bottle which held 100 ounces, whilst the compounder had thought that it held only 80 ounces. The arrangement between the Birmingham Insurance Committee and panel chemists does not recognise the use of such stock mixtures, and requires that each prescription shall be separately dispensed. The vendor was fined. J. F. LIVERSEEGE.

ANNUAL REPORT OF THE BOROUGH ANALYST FOR THE METROPOLITAN BOROUGH OF STEPNEY FOR THE YEAR 1920.

MILK.—Roughly, one sample in seventeen was adulterated, compared with one in six in 1918, and one in twelve in 1919. Notwithstanding this improvement, a simple calculation shows that there is still a great deal of tampering with milk in the borough.

The average composition of the 858 samples of milk analysed during the year was: Total solids, 12·40; solids, not fat, 8·81; and fat, 3·59 per cent. These figures, which include those of grossly adulterated samples, entirely refute the statement, frequently made in court and elsewhere, that farmers have difficulty in producing milk to satisfy the minimum limits prescribed by the Sale of Milk Regulations. One of the samples contained 35 grains per gallon of boric acid. Proceedings were taken under the Public Health (Milk and Cream) Regulations, and the vendor was fined £100.

BORIC ACID IN BUTTER AND MARGARINE.—Of 89 samples of butter analysed, 46·3 per cent. were free from preservative, 26·2 per cent. contained 0·1 per cent. of boric acid, 17·5 per cent. contained 0·2 per cent., 5 per cent. contained 0·3 per cent., and 5 per cent. contained 0·4 per cent. These figures show that butter can be sold free from preservative, and I consider that preserved butter should be required to have a declaratory label similar to that required in the case of preserved cream.

Seventy-three samples of margarine were analysed, 10·9 per cent. of which were free from preservative, whilst 8·7 per cent. contained 0·1 per cent. of boric acid, 43·5 per cent. contained 0·2 per cent., 32·5 per cent. contained 0·3 per cent., and 4·4 per cent. contained 0·4 per cent.

HERBERT HAWLEY.

* Cf. Beach, Needs, and Russell, ANALYST, 1921, 279.

DEPARTMENT OF SCIENTIFIC AND INDUSTRIAL RESEARCH.

REPORT OF THE FOOD INVESTIGATION BOARD FOR THE YEAR 1920.*

FISH PRESERVATION COMMITTEE.—An interim report on the method of preserving fish by freezing them in brine has been published. Further results obtained in the investigation of the freezing process, and in the rate of penetration of salt from brine of definite concentrations, have also been published (Adair, *Biochem. J.*, 1920, 14, 762).

MEAT COMMITTEE.—It is commonly accepted that the formation of lactic acid plays an important part in the ripening of meat and in the changes undergone by flesh on prolonged storage. Experiments have shown that continuous exposure to the freezing-point will produce a non-irritable condition in frog's muscles, and that too without the production of lactic acid, if the muscles be kept in air; but that, if kept in nitrogen, the loss of irritability is much less rapid, and lactic acid is formed. Slow autolysis takes place during the storage of meat, but whether lactic acid is formed has not yet been determined (Foster and Moyle, *Biochem. J.*, 1921, 15, 334).

A simple technique for the cultivation of anaerobic bacteria has been devised, and the spore formation of these bacteria investigated. The conditions under which mould fungi develop upon meat have also been studied (Brooks and Kidd, *ANALYST*, 1921, 292).

ENGINEERING COMMITTEE.—Investigations of the thermal conductivity and specific heat of insulating materials, on the thermodynamics of liquid carbonic acid (*Proc. Roy. Soc.*, 1920, 98A), and on the question of the measurement of humidity have been carried out.

FRUIT AND VEGETABLE COMMITTEE.—Experiments on the storage of English apples have been made with a view to determining the most suitable conditions. The method of "gas storage" approximately doubled the storage life of the varieties tested. Experiments on the preservation of fruit by freezing for jam-making have shown that the only fruits which can be satisfactorily kept in this way are those which only contain a peroxidase, but no oxygenase or catechol substance (Onslow, *ANALYST*, 1921, 292). The following fruits could therefore be kept in a frozen condition for several months in air for jam-making: Strawberries, raspberries, black currants, red currants, and gooseberries; whilst cherries and plums could only be kept unaltered in a frozen condition in an atmosphere free from oxygen. The effect of an excess of carbon dioxide on stored apples is very similar to that of low temperature. The amount of sucrose and pectin present in the juice decreases as the concentration of carbon dioxide increases; a similar increase also takes place in the ratio of fructose to dextrose, but the acidity is but little affected.

Mangin's observations on the existence of pectic compounds in three forms have been confirmed, and methods have been worked out for the extraction of each form of pectin. A new method of estimating pectin, based on its precipitation from an aqueous extract by means of alcohol, has been devised, and it has been established that more pectin can be obtained from fruit tissue by the action of heat and acid than is originally present in the soluble form. At a certain stage of over-ripeness the pectic acid seems to disappear. Experiments are being made to ascertain whether the enzyme pectase is concerned in the production of pectic acid in the fruit.

OILS AND FATS COMMITTEE.—Investigations of the syntheses of isomeric oleic acids have been continued, and a further examination has been made of the unsaturated fatty acids of reduced linseed oil (iodine value, 96). It has been found that the main constituent of the unsaturated fatty acids is oleic acid, and evidence

* Copies of the Report can be obtained from H.M. Stationery Office, Imperial House, Kingway, W.C. 2. Price 1s. net.

has been obtained that an olein results from the reduction of the glycerides of linolic acid.

The constitution of the synthetic fats obtained by the interaction of methyl glucoside or mannitol and olive oil has been studied. In the case of the methyl glucoside compounds condensation, resulting in the union of one oleyl residue with the carbohydrate chain, takes place first, and, later, the carbohydrate chain loses one molecule of water, and the synthetic fat anhydromethyl glucoside mono-oleate is obtained. In the case of the compounds of mannitol and olive oil two oleyl residues take part in the reaction.

Sterols and the Iodine Value.—The abnormally high iodine value and low saponification value of yeast fat have been proved to be due to the presence of ergosterol (a sterol believed by Tanret to be characteristic of the lower plants). In determining the iodine value of sterols by means of Wijs' reagent a considerable amount of substitution occurs, owing to the influence of the glacial acetic acid. This substitution effect is particularly noticeable when a condensed ring nucleus containing one double bond reacts with the Wijs' solution. Hence, in the case of a fat which contains an appreciable amount of sterol, the Hübl and Wijs' iodine values will differ to some extent, and the former value will more accurately estimate the degree of unsaturation of the fat. An attempt is being made to apply the difference between the iodine values obtained by the two methods under controlled conditions as a means of estimating the sterol in a fat (MacLean and Thomas *Biochem. J.*, 1921, 15, 319).

CANNED FOODS COMMITTEE.—Results obtained by W. Savage have proved that putrefying meat has little obvious effect upon the health of animals, and the conclusion is drawn that cases of poisoning from tinned meat are due neither to putrefactive bacteria nor to their products, but to the presence of some specific and highly toxic organism, such as Gaertner's bacillus.

The chemical changes occurring in untreated and canned herrings have been studied. Although it is improbable that autolytic enzymes ever act alone in untreated herrings, there is reason to believe that without the intervention of bacteria they might bring about an improvement in quality. On the other hand, deterioration is probably due to the further action of bacteria. In the case of tinned herrings, the bacterial product might arise before or after canning. Since trimethylamine is not, so far as is known, produced by enzymic action, and appears at an early stage in decomposing herrings, its estimation should throw light upon the problem. A new process of estimating it in the presence of other volatile bases has been based on Foreman's alcohol titration method (*ANALYST*, 1920, 45, 380), and is being used in investigating the question.

Estimations of the total acid radicles, volatile organic bases, and amino-acids (Foreman, *loc. cit.*) in canned and fresh herrings have shown that the former contain about twice the quantity of amino-acids, sixteen times the volatile acids, six to seven times the organic acid radicles, and five times the "net acidity" present in the latter. The small increase in amino-acids indicates that proteolysis only proceeds to a small extent in the canning and maturation. It seems probable that one or more highly nitrogenous constituents of the herring is destroyed, with the production of volatile bases.

Fresh herrings are distinctly acid in reaction, and it is probable that the changes produced in canning are largely due to the action of the acids under the influence of heat on the complex compounds present. Tinned herrings contain appreciable amounts of tin, as well as a small amount of iron, and it is possible that compounds of these metals may have a catalytic effect in continuing at the ordinary temperature the changes occurring in the canning process.

U.S.A. SERVICE AND REGULATORY ANNOUNCEMENTS.

MISBRANDING OF EGG SUBSTITUTE (*U.S. Dept. Agric. Bureau of Chemistry, Service and Regulatory Announcements*, 1921, N.J. 9290).—Analysis of a sample of "Eggno," stated on the label to be "an excellent substitute for eggs . . . to be used for baking and cooking purposes," showed that it consisted of rice starch, skim milk powder, and milk casein or albumin, artificially coloured with a coal-tar dye, and possibly containing a small amount of egg powder. Baking tests made by the Bureau showed that cakes made with the product were no better in volume or quality than cakes made with water, and were in no measure comparable in volume or quality with cakes made with an equivalent amount of eggs, as claimed on the label. The case was heard on December 9, 1920, in a District Court of the United States, and the defendants were convicted and fined. Notice of appeal was given, and the case is pending.



ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Proportion of Fat in Casein. D. M. Gangolli and A. N. Meldrum. (*Dept. Industries, Bombay Presidency, Bull. No 4*, 1921.)—A method recommended for the estimation of fat in casein consists in placing 15 c.c. of $\frac{N}{2}$ sodium hydroxide solution in a separating funnel, adding 1 grm. of the powdered casein gradually, and shaking the mixture until the casein is dissolved completely. The mixture is then extracted with ether, the ethereal solution evaporated, and the residue of fat weighed. Solution of the casein in hydrochloric acid tends to give high results for the fat, owing to partial hydrolysis of the casein, with the formation of substances which are soluble in ether. The average quantity of fat in casein is 0.25 per cent., but is usually more than this in the case of Indian casein. The colour of casein depends largely on the amount of fat present; pure casein may be prepared by washing the crude product with alcohol, and then extracting it repeatedly with petroleum spirit. Ether should not be used, as it is liable to contain peroxide, which may contaminate the casein.

W. P. S.

Separation and Identification of "Sandy" Crystals in Ice Cream. H. F. Zoller and O. E. Williams. (*J. Agric. Research*, 1921, 21, 791-795.)—It is generally assumed that the "sandiness" of ice cream is due to the separation of sucrose crystals, although no scientific evidence is given in support of this statement. A number of "sandy" ice creams were allowed to melt at the ordinary temperature, the "sand" was separated by means of the centrifuge, washed with water saturated with sucrose and lactose, and with this solution containing 50 per cent. of acetone, then dried on a Buchner funnel, and, finally, by heat in a water oven. Microscopic examination, crystallographic measurements, and analysis by the Munson and Walker method identified the "sand" as α -lactose (lactose monohydrate).

T. J. W.

Estimation of Starch by means of Taka-Diastase. E. Horton. (*J. Agric. Science*, 1921, 11, 240-257.)—The results obtained in the estimation of starch in wheat by the use of taka-diastase varied according to the sample and quantity of the enzyme employed. These variations led to an extensive investigation into the action of this enzyme upon pure potato starch. In the method applied, with modifications, the starch, gelatinised in boiling water, was cooled to 38° C., when toluene and the enzyme were added, and the temperature maintained at 38° C. for sixteen to twenty-four hours. After the mixture had been heated to 100° C. for fifteen minutes, sodium fluoride was added, and the solution was cooled, treated with basic lead acetate solution, diluted to definite volume, and filtered. Excess of lead was then removed by the gradual addition of powdered sodium carbonate, and, after filtration, the rotatory and reducing powers were determined, and the starch calculated from the dextrose and maltose found. The purity of the starch employed was ascertained by the use of the same method with malt diastase instead of taka-diastase. The factors studied included the following: Source of enzyme, age of enzyme, age of *Aspergillus oryza* culture from which the enzyme was prepared, variation in amount of enzyme used, time of hydrolysis, addition of yeast extract or of malt diastase to the enzyme, and variation in the clarifying agents used. The results obtained varied between 86 and 97·8 per cent. of starch, and, although great differences in the dextrose-maltose ratio were found, a large proportion of the results was within the limits of 91 and 96 per cent. The conclusion is drawn that the taka-diastase method is unreliable, and test experiments should be made upon pure starch with every sample of enzyme before and during use. It is possible that the discrepancy observed is due to the persistence of dextrin, but this has not been proved.

T. J. W.

Examination of Sugar Crystals by Projection. G. P. Meade. (*J. Ind. Eng. Chem.*, 1921, 13, 712.)—A small amount of the sugar to be examined is placed in a 40 mm. Petri dish, a few c.c. of alcohol saturated with sugar are added, and the crystals are separated and distributed over the dish. The latter is then fitted in the holder of the projection apparatus. The author uses a Bausch and Lomb balopticon with a lens of 8-inch focus, the projection lens having a diameter of 1·6 inches; the instrument is provided with a vertical attachment, in order that the object to be projected may be placed in a horizontal position. A magnification of ten diameters is obtained when the centre line of the projection lens is 7 feet from the screen. The latter consists of a smooth plaque of plaster of Paris about 18 inches square, and ruled into squares of various sizes (for example, 4, 6, 8, 10, and 12 mm. squares), so that, allowing for the magnification, the size of the crystals may be measured.

W. P. S.

Revision of the Optical Method for Analysing Mixtures of Sucrose and Raffinose. C. A. Browne and C. A. Gamble. (*J. Ind. Eng. Chem.*, 1921, 13, 793-797.)—The change in the Herzfeld divisor of the Clerget formula for estimating sucrose from 142·66 to 143·0 has necessitated a revision of the Creydt formula for analysing mixtures of sucrose and raffinose. On the basis of the invert factor—0·33 for sucrose and +0·514 for raffinose, and 1·852 as the ratio of the specific rotation of sucrose to that of raffinose, the following formulæ give the percentages of sucrose (*S*)

and raffinose (*R*) in a mixture when the sucrose normal weight of the sample is taken for the estimation :

$$S = \frac{0.514P - P'}{0.844} \text{ at } 20^{\circ} \text{ C., and } R = \frac{0.33P + P'}{1.563} \text{ at } 20^{\circ} \text{ C.}$$

P is the direct polarisation, and *P'* the invert polarisation of the solution. General formulæ applicable at all temperatures are :

$$S = \frac{P(0.478 \times 0.0018t) - P'(1.006 - 0.0003T)}{(0.908 - 0.0032t)(1.006 - 0.0003T)}, \text{ and}$$

$$R = \frac{P(0.43 - 0.005t) + P'(1.006 - 0.0003T)}{(1.681 - 0.0059t)(1.006 - 0.0003T)},$$

where *T* is the temperature of the direct polarisation, and *t* that of the invert polarisation.

W. P. S.

Detection of Tartaric Acid in Wines. L. Mathieu. (*Ann. Chim. anal.*, 1921, 3, 270-271).—Free tartaric acid in wine may be separated from potassium hydrogen tartrate and calcium tartrate by means of amyl alcohol. Twenty c.c. of wine are shaken with about an equal volume of amyl alcohol, and, after separation, the clear layer of amyl alcohol is removed and shaken with an equal volume of water. The aqueous layer is then separated and evaporated to dryness to remove the last traces of amyl alcohol, which causes the precipitation of calcium lævo-tartrate, and the residue is dissolved in water (5 to 6 c.c.) and treated with a drop of calcium chloride solution (150 grms. $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ and 40 grms. NH_4Cl per litre) and 2 c.c. of ammonium lævo-tartrate free from dextro-tartrate (20 grms. per litre of 20 per cent. alcohol). In the presence of tartaric acid a precipitate of calcium racemate is formed immediately; 0.5 gram. of free tartaric acid per litre of wine may be detected by the above method, and smaller amounts by using a larger sample of the wine.

R. G. P.

Chemical Composition of Arachis Oil. G. S. Jamieson, W. F. Baughman, and D. H. Brauns. (*J. Amer. Chem. Soc.*, 1921, 43, 1372-1381).—Two specimens of arachis oil expressed in the laboratory from nuts of the white Spanish type and the Virginian type had iodine values of 90.1 and 94.8 respectively. The mixed fatty acids yielded no linolenic hexabromide, but a linolic tetrabromide. The proportions of linolic and oleic acids were calculated from the percentages of bromine in the bromides. The saturated fatty acids were separated by the lead-ether method, and fractionated by distillation of their methyl esters under reduced pressure, corrections being made from the iodine and saponification values for the amounts of unsaturated fatty acids in each fraction. By these means the two oils were found to have the following percentage composition :

	Oleic Acid.	Linolic Acid.	Palmitic Acid.	Stearic Acid.	Arachidic Acid.	Lignoceric Acid.	Unsaponifiable Matter.
Oil from Spanish nuts ...	52.9	24.7	8.2	6.2	4.0	3.1	0.2
Oil from Virginia nuts ...	60.6	21.6	6.3	4.9	3.3	2.6	0.3

No hypogæic acid or behenic acid was present.

Detection of Tea-Seed Oil in Olive Oil.* J. Dybowsky and E. Millia. (*Comptes rend. Acad. Agric. de France*, 1920, 6, 577-581; *Inter. Rev. Sci. Agric.*, 1921, 11, 1193-1194.)—The presence of 10 to 20 per cent. of tea-seed oil in olive oil may be detected by shaking 20 grms. of the oil for one minute with 6 drops of sulphuric acid; the mixture is then treated with 9 drops of nitric acid, shaken for one minute, heated at 100° C. for five minutes, and finally cooled to 10° C. After two hours at the latter temperature the mixture remains liquid if 20 per cent. of tea-seed oil is present; if the original oil contained 10 per cent. of tea-seed oil the mixture solidifies, but the mass is not as hard as that obtained with pure olive oil. A characteristic reaction, which will detect as little as 5 per cent. of tea-seed oil, consists in shaking 4 c.c. of the oil for thirty seconds with a mixture of 5 c.c. of pure sulphuric acid, 3 c.c. of nitric acid, and 3 c.c. of water; the whole mixture is kept at 5° C. for five minutes and observed after a further fifteen minutes. Pure olive oil gives a straw coloration and remains clear, pure tea-seed oil becomes turbid and sooty-black, whilst olive oil containing 5 per cent. of tea-seed oil gives a dark straw-coloured, turbid mixture. These colorations are observed in the oily layer; the acid layer is colourless in all cases. W. P. S.

Soya-Bean Oil. C. R. Fellers. (*J. Ind. Eng. Chem.*, 1921, 13, 689-691.)—The quantity of oil in soya beans varies considerably; in twenty-six varieties of the bean examined, the amount ranged from 14.6 to 25.6 per cent.; the quantity of protein varied from 35.7 to 44.3 per cent. The sp. gr. of the oil is practically constant at 0.925, and the refractive index at 25° C. varies but little from 1.474; the iodine value ranges from 123.2 to 132.3, and the saponification value from 190 to 195. There appears to be some relation between the colour of the beans and the drying property of the oil, dark beans yielding oil of rather poor drying power. W. P. S.

Chemical Investigation of Mutton-Bird Oil.—I. C. L. Carter. (*J. Soc. Chem. Ind.*, 1921, 40, 220r.)—The oil extracted from the stomach of the Australasian petrel (*Æstrelata lessona*) is a fairly thin, straw-coloured, non-drying oil, closely resembling sperm oil in chemical characteristics. The oil contains 38 per cent. of alcohols, chiefly cetyl alcohol, and 61 per cent. of fatty acids, which are chiefly oleic acid with some acids of the linolic or linolenic series. At 6° C. spermaceti separates out, and at 0° C. the oil solidifies. The chief constants are: Sp. gr. at 15° C., 0.884; free fatty acids, 2.23 per cent.; alcohols, 38.4 per cent.; saponification value, 119.6; iodine value, 130 (Wijs), 108 (Hübl); viscosity, 0.3724; m.-pt. of alcohols, 31° to 31.5°; saponification value of acetate, 189; and iodine value of fatty acids, 150 (Hübl), 178 (Wijs). (Cf. *ANALYST*, 1911, 36, 290.) H. E. C.

Sweet Fennel Oil. C. E. Sage. (*Perf. Essent. Oil Rec.*, 1921, 12, 46; *Chem. Abstracts*, 1921, 15, 1597.)—Samples of this oil obtained on the market are in some cases apparently derived from the whole Spanish plant instead of from the fruit only. They are distinguished from the genuine oil by the almost entire absence of anethole,

* The tea-seed oil giving these reactions had probably not been refined.—EDITOR.

and are suitable for flavouring purposes only. Two samples examined showed the following characteristics: Sp. gr. at 15.5° C., 0.9203 and 0.9340; n_D^{25} , 1.4945 and 1.4980; $[\alpha]_D$, +46 and +24; solubility in 90 per cent. alcohol, 1 in 1; and both remained liquid on cooling to -15° C. It is suggested that such products should be designated "plant oils," in contradistinction to the "fruit oils" usually manufactured.

T. J. W.

Identification of Ouabain and Strophanthin. A. Richaud. (*J. Pharm. Chim.*, 1921, [vii.], 24, 161-166.)—For the identification of these glucosides the following properties serve: Ouabain forms a pure white powder of nacreous appearance, and crystallises in rectangular plates. It dissolves in 150 parts of water at 15° C., giving an absolutely colourless, transparent solution, which has a slight bitter taste, and gives no lasting froth when shaken. If a few crystals of resorcinol and then a few of ouabain are added to 4 to 5 c.c. of concentrated hydrochloric acid in a test-tube, and the latter is heated to 60° to 70° C. in a water-bath for a few moments, no coloration appears. Strophanthin is usually a dirty white or very pale yellow powder, and is amorphous or crystallised in spangles, often arranged radially; 1 part dissolves in 40 to 43 parts of water at 15° C., giving a solution which is not absolutely colourless or transparent, has a very marked bitter taste, and yields a persistent froth when shaken. When treated with hydrochloric acid and resorcinol, as described above, strophanthin gives a pink coloration.

T. H. P.

Microchemical Tests for Saccharin and its Salts. Denigès. (*Bull. Soc. Pharm. Bordeaux*, 1921, No. 2; *Ann. Chim. anal.*, 1921, 3, 273-275.)—The silver salt of saccharin is produced by treating on a microscope slide less than 1 mgrm. of a soluble salt of saccharin, such as the ammonium salt ("sucramine") or the sodium salt ("sucrose"), with a drop of ammoniacal silver nitrate solution (3 per cent.). The crystals are of characteristic appearance, and serve for the microchemical identification of saccharin salts. Saccharin itself, which is almost insoluble in water, is first dissolved in a drop of ammonia, and the solution evaporated to dryness on the microscope slide. The crystals of saccharin obtained by the treatment of the sodium or ammonium salt with dilute sulphuric acid (10 per cent.), or with concentrated hydrochloric acid, are also of characteristic appearance. The sodium salt of saccharin may be detected by the formation of eubic crystals of sodium chloride produced on acidification with hydrochloric acid, whilst the ammonium salt yields prismatic crystals of hydrogen ammonium tartrate when treated with sodium hydrogen tartrate.

R. G. P.

Derivatives of Sulphur in Commercial Salvarsan.—II. H. King. (*J. Chem. Soc.*, 1921, 119, 1415-1420.)—The substance $C_6H_5O_6NSAs$, obtained as a by-product in the reduction of 3-nitro-4-hydroxy-5-sulphinophenylarsinic acid with sodium hyposulphite, has now been proved to be the sulphonic acid of phenylarsenious acid, and not the sulphinic acid of the phenylarsinic acid. Hence, the amorphous arsenobenzenes containing the sulphinic acid group in commercial salvarsan (ANALYST, 1921, 407) are in reality identical with the corresponding sulphonic acids,

and the slight differences in analytical figures and toxicity are those of amorphous substances prepared under different conditions. The principal sulphur-containing impurity in commercial salvarsan is 3 : 3'-diamino-4 : 4'-dihydroxy-5-sulphoarsenobenzene monohydrochlorides associated with varying proportions of "salvarsan sulphate."

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Detection of Secretion Disturbances in Milk. G. Koestler. (*Mitt. Lebensm. Hyg.*, 1920, 11, 154-169; *Chem. Abstracts*, 1921, 15, 1581.)—Very slight pathological changes affect the milk, causing an increase in the serum-soluble nitrogen, chlorine, and sodium, and a decrease in the lactose, potassium, and phosphoric acid. The extent of the disturbance may be measured by calculation of the "chlorine-sugar" number from the formula $100 \left(\frac{\text{chlorine per cent.}}{\text{lactose per cent.}} \right)$. For normal milks this value was found to be 0.49, whilst for a pathological milk having a normal appearance the value was 15. The ratio of chlorine to phosphoric acid in the ash may be employed in a similar manner for the detection of pathologically abnormal milk. The abnormal milks examined showed a normal depression of the freezing-point.

T. J. W.

Estimation of Small Quantities of Phosphates in Biological Products. G. Denigès. (*Comptes rend. Soc. Biol.*, 1921, 84, 825; *J. Pharm. Chim.*, 1921, [vii.], 24, 184-186.)—The reagents employed in this method are: (a) Sulphomolybdic reagent, prepared by mixing equal volumes of 10 per cent. ammonium molybdate solution and concentrated sulphuric acid; (b) stannous chloride: 0.1 gm. of thin tinfoil is treated in a test-tube with 2 c.c. of hydrochloric acid and 1 drop of 3 to 4 per cent. copper sulphate solution, the liquid being warmed gently until the metal dissolves, and the solution diluted to 10 c.c. with water, allowed to cool, and decanted from any deposit. Before the test is applied, the organic matter must be destroyed in either of the two following ways: (1) The substance is incinerated with one-third or one-fourth of its weight of calcined magnesia free from phosphates, the residue taken up in water containing a little sulphuric acid, and the liquid diluted to a known volume after prolonged boiling to transform pyro- or meta-phosphoric ions into phosphoric ions. (2) By means of nitric and sulphuric acids: With blood, for instance, 1 gm. or 1 c.c. is mixed in a porcelain dish about 8 cm. wide with 1 c.c. of nitric acid, 1 c.c. of sulphuric acid, and, drop by drop, 30 to 40 drops of 90 to 95 per cent. alcohol. When effervescence ceases, the liquid is covered with a funnel fitting inside the dish, which is heated gently on gauze or asbestos card. If the liquid darkens when white sulphuric acid fumes appear, it is heated again with a few drops of nitric acid, these operations being continued until the solution remains uncoloured, when all the nitric acid is expelled. The liquid is then cooled, diluted with 10 to 12 c.c. of water, boiled to one-half its volume, and finally made up to 200 c.c. To 5 c.c. of the solution obtained by either of these methods are added 4 drops of the above sulphomolybdic reagent, and, after mixing, 2 to 4 drops of the stannous chloride solution, which must be freshly prepared. After further mixing, the tube is left for ten

minutes, and the blue coloration of its contents then compared with those of the liquids in a series of tubes similarly prepared from 5 c.c. of solutions containing from 0.001 to 0.012 gm. of P_2O_5 per litre. This reaction is most sensitive with solutions containing between 0.0005 and 0.010 gm. of phosphorus per litre. T. H. P.

Rose Bengal as a General Bacterial Stain. H. J. Conn. (*N.Y. Agric. Expt. Stat. J. Bact.*, 1921, 6, 253-254; *Chem. Abstracts*, 1921, 15, 1551.)—Rose bengal readily stains bacterial protoplasm, but has little affinity for slime and debris surrounding the organism. It is of particular value for staining slime-forming bacteria, such as *B. radicumicola*. The stain, which remains in good condition for several months, is prepared by dissolving 1 gm. of rose bengal in 100 c.c. of 5 per cent. phenol solution. T. J. W.

Detection of Manurial Pollution in Milk by the Anaerobic Spore Test. J. Weinzirol. (*Amer. J. Public Health*, 1921, 11, 149-152; *Chem. Abstracts*, 1921, 15, 1581-1582.)—From 0.5 to 1 gm. of melted paraffin wax is poured into a test-tube, which is then plugged with cotton-wool and sterilised. Five c.c. of the milk under examination are run from a sterile pipette into each of five tubes containing the wax, and the whole are heated to 80° C. for ten or fifteen minutes. The tubes are then incubated at 37° C. for three days, when the presence of anaerobes is indicated by the evolution of gas, which lifts the wax plugs. Two positive results out of five indicate excessive pollution. The results obtained suggest that the number of infected samples detected is probably too low, owing to caramelisation of the lactose present, but a higher proportion is found by the addition of lactose to the milk. The method is considered to yield results of equal value to those obtained by the *B. coli* test. T. J. W.

Notes on *B. coli* and *B. aerogenes*. M. Levine. (*Amer. J. Public Health*, 1921, 11, 21-23; *Chem. Abstracts*, 1921, 15, 1740.)—The growth of *B. coli* is inhibited in 0.5 per cent. peptone medium containing 1 part per 200,000 of crystal violet, or 1 part per 1,000,000 of brilliant green. The concentration of the peptone in culture media has a decided influence upon the inhibiting action of dyes upon bacterial growth. The growth of *B. coli* is stimulated by a bile-salt concentration of less than 0.5 per cent., but is partially inhibited when the concentration is increased to 0.7 or 1.0 per cent. The production of both liquid and solid media which will inhibit the growth of *B. coli*, and not that of *B. aerogenes*, appears practicable. T. J. W.

Souring of Beef caused by *Bacillus megatherium*. H. Bunyea. (*J. Agric. Research*, 1921, 21, 689-698.)—The condition known as "sour beef" is due to an acid fermentation, which occurs when the meat is stored, while still warm, under conditions which do not allow it to cool. An organism was isolated from the interior portion of a sample of such meat, and was found to correspond closely in morphology and cultural characteristics with *B. megatherium* var. *Ravenellii* (from soil) and *B. megatherium* var. *de Bary* (from cabbage infusions). It was a round-ended, chain-forming, spore-bearing, aerobic, slightly motile rod, from 2 to 5 μ long and 2 μ wide

During sporulation the organisms became symmetrically swollen; the spores were equatorial. Colonies of twenty-four hours' growth were densely opaque at the centre, whilst the periphery was granular, and had the appearance of irregular circles. When grown on agar and potato, the organisms formed yellowish growths, resembling those of *B. subtilis*. This bacillus, like *B. megatherium* var. *de Bary*, produced the "sour beef" condition in sterile specimens of raw beef, the optimum temperature being about 37° C. The offensive putrefactive odour was due to propionic acid, which could be isolated by extracting the sour beef with 5 per cent. sulphuric acid and distillation of the filtered extract with steam. The organism was not pathogenic to guinea-pigs, and did not produce any appreciable amount of toxin in ordinary media or raw beef under laboratory conditions.

Serological Method for Detection of Infected Foods. J. Bronfenbrenner and M. J. Schlesinger. (*Proc. Soc. Exp. Biol. Med.*, 1921, 17, 24-25; *Chem. Abstracts*, 1921, 15, 1766.)—The whole sample is finely chopped, and an extract prepared, concentrated to a small volume, and tested against a set of specific immune sera. The presence of *B. botulinus* protein in 20 gm. samples of inoculated food was detected when the proportion of toxin was so small that a mouse weighing 15 to 20 grms. required 7 grms. administered by the mouth, or 1.3 grms. by injection, to show definite symptoms. If a collection of specific sera of high titre is available, the above procedure may be completed within twenty-four hours. T. J. W.

Colour Reaction Common to Antiscorbutic Extracts and Hydroquinone. M. Bezssonoff. (*Comptes rend.*, 1921, 173, 466-468.)—Folin's reagent for phenols is prepared by boiling for two hours a mixture of 100 grms. of sodium tungstate, 20 grms. of phospho-molybdic acid, and 50 c.c. of concentrated phosphoric acid, in a litre of water. When used in the presence of an excess of sodium carbonate it gives a blue coloration with mono-, di-, and triphenols and their derivatives, and with many vegetable substances containing or not containing vitamin C. A modified reagent, in which the quantity of phosphoric acid is reduced to a third, and to which, without boiling, an equal volume of *N*-sulphuric acid is added, gives a slate-grey coloration, changing to blue, with vegetable juices (orange, lemon, tomato, grape, cabbage) known to have pronounced antiscorbutic power. On the other hand, extracts of seeds (oats, turnip) without antiscorbutic power do not give the coloration. The plum, which is an anomaly among fresh vegetable products by reason of its being devoid of antiscorbutic power, does not give a blue coloration, but a yellowish-brown coloration, persisting for three hours; whereas the peach, belonging to the same genus as the plum, gives the blue coloration. Causes which diminish or destroy the antiscorbutic power, such as exposure to the air or boiling, also reduce or inhibit the blue coloration. Potato juice extracted in the presence of acid gives a distinct blue coloration; whereas the juice extracted without acid (weak antiscorbutic power) gives no coloration, or only a faint one. Cow's milk, which has only a slight antiscorbutic power, does not give the reaction, except a fugitive coloration at the moment of adding the reagent in the case of chilled milk. Urine from a man whose diet has been sufficiently rich in the antiscorbutic principle

gives a positive reaction. It is suggested that the reaction is probably due to an unstable radicle, probably a polyphenol, which readily becomes detached from vitamin C in its solutions. The reactions given by this reagent with phenols are essentially different from those obtained with Folin's reagent. Monophenol, salicylic acid, and tyrosin do not give any reaction; whilst tannin and guaiacol give a brownish-yellow coloration. The only compound tried which gives the blue coloration of Folin's reaction is hydroquinone. Pyrocatechol and catechol give a reddish violet coloration changing to blue, whilst phloroglucinol gives no coloration. The complex giving the reaction can be separated in soluble monoclinic crystals having a composition corresponding with the formula— $(17\text{WO}_3)(\text{MoO}_3)(\text{P}_2\text{O}_5\text{H}_2\text{O})21\text{H}_2\text{O}$. This differs from the complex in Folin's reagent in having a lower proportion of molybdenum trioxide to that of tungsten trioxide.

AGRICULTURAL ANALYSIS.

Estimation of Clay in Heavy Soils. A. F. Joseph and F. J. Martin. (*J. Agric. Science*, 1921, 11, 293-303.)—The Sudan method, as devised by Beam (*Cairo Scientific J.*, 1911, 107-119), is recommended as preferable to the English or American methods generally used. One hundred grms. of soil are dried at 100° C., and sifted through a 2 mm. circular mesh. The portion passing through is again sifted through a 1 mm. mesh. Five grms., or less, of the portion passing through are shaken for two hours with 100 c.c. of 0.2 per cent. sodium carbonate solution. The mixture is transferred to a beaker, diluted with water to a height of 10 cm., well mixed, and allowed to stand for sixteen hours, when the clay suspension is decanted, and the residue treated with 0.05 per cent. sodium carbonate solution, left to stand, and again poured off, this being repeated six to nine times. A final decantation is made with water, and the residue dried at 100° C. and weighed. The silt is removed by washing the dried residue into a beaker, and after leaving it for seven and a half minutes in a 10 cm. column of water, decanting the liquid, washing the remainder back into the original beaker, drying and weighing it, subsequently separating it into "coarse sand" and "fine sand" by sifting it through a mesh of 0.2 mm. The "coarse sand" is added to the portion already obtained not passing the 1 mm. sieve. A centrifugal method is also described which requires constant attention, but allows of results being obtained in much less time. In both cases the time required is considerably less than with either the American or English methods. The soil particles are classified in the three methods according to the following table:

		METHOD.		
		English.	American.	Sudan.
d stones	...	Above 1 mm.	1 to 2 mm.	Above 2 mm.
Coarse sand	...	1 to 0.2 mm.	1 to 0.05 mm.	2.0 to 0.2 mm.
Fine sand	...	0.1 to 0.04 mm.		0.2 to 0.02 mm.
Coarse silt	...	0.04 to 0.01 mm.	0.05 to 0.005 mm.	0.02 to 0.002 mm.
Fine silt	...	0.01 to 0.002 mm.		
Clay	...	Below 0.002 mm.	Below 0.005 mm.	Below 0.002 mm.

T. J. W.

ORGANIC ANALYSIS.

Detection of Oxalic and Citric Acids. Polonovski. (*J. Pharm. Chim.*, 1921, [vii.], 24, 167-168.)—Oxalic acid may be detected as follows: The mixture having been decomposed in the usual way by means of sodium carbonate, 2 to 3 c.c. of the alkaline filtrate are acidified with acetic acid, and the solution treated with 1 c.c. of saturated calcium fluoride solution; under these conditions oxalates alone are precipitated. If the mixture is free from fluorides, the calcium fluoride added may be replaced by calcium sulphate solution. To detect citric acid, the dissolved or powdered substance is treated at 50° to 60° C. with potassium permanganate solution until it remains pink, and is then heated gently with 1 c.c. of bromine water until it becomes decolorised; in presence of citric acid, perbromoacetone is formed, this being easily recognisable by its pungent action on the mucous membranes of the eyes.

T. H. P.

Greasiness of Fatty Substances. P. Woog. (*Comptes rend.*, 1921, 173, 303-306.)—The author considers that the greasiness of fatty substances depends to some extent on the tendency of the molecules to coalesce (*coincement*). As this tendency should become more pronounced as the molecules increase in dimensions, the molecular volumes have been determined microscopically for a number of fatty and mineral oils and for glycerol. It is found that the molecular volumes of the fatty oils are distinctly higher than those of the mineral oils of corresponding viscosity. For mineral oils, the greasiness runs closely parallel with the molecular volume; the dictum stating that, for equal viscosities, the most greasy oil is that of lowest density, is thus explained, since in a homologous series the viscosity increases with the molecular volume. For example, the following results were obtained:

	Sp. gr. at 15° C. (<i>D</i>).	Mole- cular Weight (<i>M</i>).	$\frac{M}{D \cdot N}$ ($N = 61 \cdot 10^{22}$).	<i>K</i> .
<i>Fatty Oils:</i>			c.c.	
Colza	0.9149	912	$1634 \cdot 14 \cdot 10^{-24}$	5.77
Arachis	0.9162	862	1542.36	5.64
Castor	0.9626	890	1515.75	5.47
Lard	0.9169	831	1485.76	5.21
Olive	0.9152	805	1441.94	5.05
Lanoline (anhydrous) ...	0.9556	793	1360.43	4.4
<i>Petroleum Oils:</i>				
Locomotive	0.9056	853	$1544 \cdot 12 \cdot 10^{-24}$	7.32
Cylinder	0.8946	748	1370.70	6.76
Spindle	0.8918	296	544.11	3.32
French neutral	0.8529	270	518.96	3.27

The temperature coefficient *K* of superficial molecular energy, determined by the formula of Eotvos, Ramsay, and Shields, is never lower than 2.12 for true lubricants.

T. H. P.

Oil-film Measurement of Fatty Oils, and Certain Phenomena of Molecular Solutions. P. Woog. (*Comptes rend.*, 1921, 173, 387-390.)—Measurement of films of various fatty oils (see preceding abstract) extended on the surface of water in a layer one molecule in thickness shows that, except in the case of castor oil, the molecules of oils appear to exhibit only slight asymmetry. Similar measurements made on the principal constituents of the oils give numbers differing little from those published by Langmuir (*J. Amer. Chem. Soc.*, 1917, 39, 1848) in support of his theory of surface tension. T. H. P.

Aschmann's Method of Determining the Iodine Value. B. M. Margosches and R. Baru. (*Chem. Zeit.*, 1921, 45, 898.)—Aschmann's method of determining the iodine value of oils by means of an aqueous solution of iodine monochloride (*Chem. Zeit.*, 1898, 59, 71) has been condemned on the ground of the reagent not being miscible with solutions of fat. Comparative experiments here described, however, show that the iodine values thus obtained after an absorption of twenty-four hours, during which the mixture is shaken at least six times, agree closely with the Hübl and Wijs values in the case of non-drying oils, and come between the Hübl and Wijs values in the case of drying oils (hemp and niger seed oils). The reagent has the advantage of stability of "titre."

Quantitative Separation of the Lead Salts of Saturated and of Less Saturated Fatty Acids. A. Seidenberg. (*J. Amer. Chem. Soc.*, 1921, 43, 1323-1336.)—A more accurate and sharp method of separating the lead salts of fatty acids than has hitherto been possible is based on their solution in three solvents—alcohol, chloroform, and ether—two of which are more volatile than alcohol and have also a greater solvent action—and in then volatilising the mixed solution until all the more insoluble salts are precipitated. In this way it is possible to remove completely the whole of the salts of the saturated fatty acids, and concordant results are obtained independently of the quantities used. The method is not applicable to oils, such as linseed oil, which contain linolenic acid, since the saturated fatty acids will contain more or less of that acid; but good results are obtained in the case of oils, such as cottonseed oil, which contain the less unsaturated fatty acids, oleic and linolic acids. The lead salts prepared from 2 to 5 grms. of mixed fatty acids are dehydrated by exposure to the air, and finally by washing with several portions (20 c.c. in all) of anhydrous alcohol, which are then decanted into a cylinder. The residual lead salts are dissolved in 20 c.c. of boiling chloroform, the solution evaporated to about 8 c.c., and, after the addition of about 60 c.c. of ether, boiled for a moment and poured into the cylinder containing the alcohol. The flask is rinsed out with the minimum quantity of hot chloroform and ether, and the total contents of the cylinder made up to 120 c.c. with ether. Air is now aspirated through this solution until the volume has been reduced to 65 or 70 c.c., after which the liquid is filtered through a Gooch crucible. The precipitate on the filter is redissolved in the smallest possible quantity of boiling chloroform, about 50 c.c. of ether added, and the solution boiled for a moment and allowed to cool. The mixture of solution and precipitate is filtered, and the precipitate washed three times with

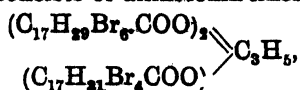
10 c.c. of ether. This process is repeated until a sufficiently pure precipitate is obtained. The filtrates are united and treated with 20 c.c. of 95 per cent. alcohol, and the volatile solvents removed by aspiration until the volume has been reduced to about 30 c.c. If the precipitate separated by filtration is not greatly discoloured when heated at 100° C., it is weighed; otherwise it is redissolved in 3 c.c. of boiling chloroform, about 20 c.c. of boiling ether added, the solution cooled and filtered, and the residue weighed. The amount of this residue, added to that of the main residue, gives the amount of the lead salts of the saturated fatty acids in the sample.

Precipitation of Solid Fatty Acids with Lead Acetate in Alcoholic Solution. E. Twitchell. (*J. Ind. Eng. Chem.*, 1921, 13, 806-807.)—The following process is recommended as being more satisfactory than the lead-salt-ether method for the separation and estimation of solid fatty acids: About 2 grms. of fatty acids from a solid fat, or 10 grms. from an oil, are dissolved in hot alcohol, and a boiling solution of 1.5 grms. of lead acetate in alcohol is added; the total quantity of alcohol used should be 100 c.c. The mixture is allowed to cool slowly, maintained at 15° C., for eighteen hours, and the precipitate then collected on a filter and washed with 95 per cent. alcohol until the filtrate remains clear when diluted with water. The precipitate is next transferred to a beaker, boiled with 100 c.c. of alcohol and 0.5 c.c. of acetic acid, the solution allowed to cool as before, and the precipitate again collected and washed, and then washed into a beaker with ether. Nitric acid is added to decompose the lead salts, the mixture is poured into a separating funnel, and the acid solution drawn off; the ethereal solution is washed thoroughly with water, then evaporated, and the residue of fatty acids dried and weighed. The amounts of solid fatty acids yielded by various oils were as follows: Cottonseed oil, 23.13; soya-bean oil, 17.01; olive oil, 10.93; earthenut oil, 16.58; lard, 40.02; tallow, 53.62; partly hydrogenated cottonseed oil, 50.62 per cent. In each case the iodine value of the solid fatty acids was below 1, except with those of the hydrogenated cottonseed oil (42.21) and lard (4.38).

W. P. S.

The "Oil Bromide" Test of Linseed Oil. T. A. Davidson. (*J. Ind. Eng. Chem.*, 1921, 13, 801-806.)—Of late years it has been the usual practice to use the fatty acids of linseed oil, and not the oil itself, for the estimation of the hexabromide value. As the drying properties of the oil are due to the glycerides of linolenic acid, and not to linolenic acid, and as there is good reason to assume that the acid exists in linseed oil as a mixed glyceride, the author considers that it is desirable to apply the test directly to the oil, as proposed originally by Hehner and Mitchell (*ANALYST*, 1898, 23, 310). The term "oil bromide" is used for the insoluble brominated glycerides, and "hexabromides" for the insoluble brominated acids. The following procedure is recommended for the estimation of the oil bromide value; the final washing with alcohol yields a product which remains a powder when dried: Four grms. of the oil are dissolved in 35 c.c. of ether in a centrifuge tube, the solution is cooled at 0° C., and 1 gm. of bromine is added slowly while the mixture is stirred. The top of the tube is rinsed down with 5 c.c. of ether, and the whole mixture maintained at 0° C. for two hours, and then submitted to centrifugal action for three

minutes at 2,000 revolutions per minute. The liquid is decanted from the precipitate, and the latter is washed twice with ether (10 c.c. each time) at 0° C. and once with alcohol, the precipitate being separated by centrifugal action after each washing. After the final washing the precipitate is dried, first in air and then at 100° C. for thirty minutes. All the ether used should be saturated previously at 0° C. with oil bromide (obtained from a former estimation). The yield of oil bromide from genuine linseed oils varies from 50 to 72 per cent.—average 60.9 per cent., a figure higher than those so far recorded. Oil bromide is an amorphous, slightly yellow solid, melting at 140° to 145° C. It contains 57 to 58 per cent. of bromine; is fairly soluble in chloroform, carbon tetrachloride, acetone, and acetic acid; slightly soluble in ether, benzene, and petroleum spirit; and practically insoluble in alcohol. It is probable that the substance consists of dilinolenin-linolin bromide,



mixed with some brominated glyceride of lower bromine content. To estimate the bromine content, a quantity of the oil bromide is mixed with 68 times its weight of alcohol, and 6.5 times its weight of sodium is added; the mixture is heated under a reflux condenser for one hour, and the resulting sodium bromide then titrated by Volhard's method. Ignition with lime yields low results for the bromine, and this method is considered to be untrustworthy for the purpose. W. P. S.

True Oxygen Absorption of Oils. II. Linolenic and Linolic Acids.

S. Coffey. (*J. Chem. Soc.*, 1921, 119, 1408-1415.)—The hexabromostearic acid obtained directly from linseed oil yields, when reduced with zinc, a mixture of α - and β -linolenic acids, from which the α -acid can be separated as hexabromide by rebromination. The mixture of the two acids was oxidised as previously described (*ANALYST*, 1921, 418); another portion of the mixture was brominated by the method of Hehner and Mitchell (*ANALYST*, 1898, 23, 310), and, after removal of the insoluble hexabromostearic acid, the residual β -acid was debrominated, and the β -linolenic acid oxidised under identical conditions. The results due to the oxidation of the separated α -linolenic acid were then obtained by difference. In applying the oxidation method to the quantitative estimation of the constituents of linseed oil the amount of α -linolenic acid is calculated from the quantity of carbon dioxide in the volatile products by multiplying by the ratio $\text{C}_{18}\text{H}_{30}\text{O}_2/\text{CO}_2$. The total oxygen absorption is due to oxidation of linolic and linolenic acids, and, since the amount of the latter is known, that of the linolic acid can be calculated. The oleic acid is calculated by difference from the total iodine value, glycerol is estimated directly, and saturated fatty acids and unsaponifiable matter found by difference. In this way a sample of linseed oil was found to have the following composition: Saturated fatty acids and unsaponifiable matter, 8.1; glyceryl radicle, 4.3; oleic acid, 5.0; linolic acid, 48.5; and linolenic acid, 34.1 per cent.

Estimation of Sodium Chloride in Soap. H. C. Bennett. (*J. Ind. Eng. Chem.*, 1921, 13, 813.)—A weighed quantity of about 5 grms. of the soap is dissolved

in 150 c.c. of hot water, and the soap and soluble fillers are precipitated by the addition of 25 c.c. of 20 per cent. magnesium nitrate solution; the hot, unfiltered solution is then titrated with standard silver nitrate solution, potassium chromate being used as indicator.

W. P. S.

Consistence of Starch and Dextrin Pastes. W. H. Herschel and C. Bergquist. (*J. Ind. Eng. Chem.*, 1921, 13, 703-706.)—The viscosity apparatus described previously by Bingham (*ANALYST*, 1914, 39, 284) is modified in its application as a plastometer; by plasticity is meant a measure of the consistence at a given time. Two side strips are fitted to the cover of the apparatus, and aid in unscrewing the cover; the plug is made in two pieces, so that the centre of a long capillary is accessible and readily cemented into position, and a ring attached to the upper part of the plug holds the latter in position. Capillaries of 1 to 1.5 mm. diameter are used, and have a length of 25 to 40 mm. Adhesives in the concentration at which they are used are mostly plastic, but differences in shear value and rigidity of different samples of the same concentration are striking and significant. Although further work on the subject is necessary, the results so far obtained indicate that the values obtained by the plastometer will show body, spreading power, and gelatinising power of starch and dextrin pastes.

W. P. S.

Effects of Chemical Reagents on the Microstructure of Wood. A. Abrams. (*J. Ind. Eng. Chem.*, 1921, 13, 786-790.)—Cross, radial, and tangential sections are cut from the wood, and these sections are placed in the depressions of a microscope cell slide; the cells of the slide are filled with the reagent to be studied, and a plain slide is placed over the cell slide, but separated from it about 1 mm. by pieces of thin cover-glass. The two are wired together (in dealing with sodium bisulphite or other substance which attacks iron wire, the slides are sealed into glass tubes with the reagent) and placed in an autoclave containing a considerable quantity of the reagent. The autoclave is heated for various lengths of time at the required pressures, and at stated intervals is cooled quickly, and a slide removed for microscopic examination. Photomicrographs are given, showing the action of various substances on wood. In general, cellulose solvents, such as copper-ammonium reagent and 75 per cent. sulphuric acid, act strongly and about equally on both the middle lamella and the cell wall. Oxidising substances (chromic acid, bleach solutions, and potassium chlorate in nitric acid) act on the cell wall, but have comparatively little effect on the middle lamella. The ordinary paper-making reagents, such as dilute sodium hydroxide solution and sodium bisulphite solution, act strongly on the middle lamella, and have much less effect on the cell wall.

W. P. S.

Smokeless Fuels. F. S. Sinnatt. (*J. Roy. Sanit. Inst.*, 1921, 42, 22-26.)—In modern gasworks there is a tendency to produce increasingly large volumes of gas at the expense of the coke, with the result that the percentage of inorganic constituents in the residue becomes excessively high. For example, cases may be cited of coke yielding 20 to 30 per cent. of ash and containing a considerable proportion of water. Smokeless fuel should have some of the following characteristics: It should

be smokeless and ignite with ease; should not be too fragile or too bulky; should not absorb water, and, if it becomes wet, should dry in a reasonable time. It should have a low ash content, and the ash should not be powdery; it must contain only a small percentage of sulphur; must be sufficiently porous for the combustion to proceed internally as well as externally, in order that the ash produced does not cover the surface and prevent combustion, but must not be too porous, or it will be fragile. No convenient method of determining the relative values of different smokeless fuels has yet been developed, but it is advisable to ascertain the proportion of volatile matter and the texture or micro-texture of the coke. The following results were obtained in the analyses of typical varieties of such fuels:

Percentage.	Lignite, Low Tempera- ture.	Carbo- coal.	Briquette Sun Fuel Company.	Hargreaves Coke.	Special Briquette.	Low Tem- perature Carbon Coke.
Water	6.44	2.03	3.10	0.60	2.60	2.42
Ash	16.85	15.66	10.48	10.70	9.00	4.70
Volatile matter ...	22.43	5.94	10.08	7.80	13.00	15.45
Volatile organic matter	15.99	3.91	6.98	7.20	10.40	13.03
Coke	77.57	94.06	89.92	92.20	87.00	84.55
Fixed carbon	60.72	78.40	79.44	81.50	78.00	79.85
Calorific value (British thermal unit per lb.)	10,280	11,570	12,740	13,180	12,400	13,550

Detection and Estimation of Coal Tar Oils in Turpentine. V. E. Grotlisch and W. C. Smith. (*J. Ind. Eng. Chem.*, 1921, 13, 791-793.)—A method for the detection of small quantities (1 to 2 per cent.) of coal tar oils, such as benzene, toluene and commercial solvent naphtha, in turpentine, consists essentially in concentrating these oils by converting the turpentine into pinene hydrochloride, and separating the latter. The oils are then sulphonated, distilled, and identified as described below. One hundred c.c. of the turpentine are cooled in a mixture of ice and salt, and then saturated with dry hydrogen chloride; when absorption of the latter is complete, the mixture is kept for one hour in a closed vessel in the freezing mixture, to obtain maximum crystallisation of the pinene hydrochloride. This compound is very soluble in both mineral and coal tar oils, and if more than 10 per cent. of the oils is present, scarcely any crystals are obtained. The crystals are separated by filtration, and the filtrate is distilled under reduced pressure (10 inches of mercury) until crystals separate out in the condenser, or until 25 c.c. of distillate are obtained. The distillate is added drop by drop to four times its volume of fuming sulphuric acid (containing 4 per cent. of free sulphur trioxide), and the mixture is then heated at about 70° C. for twenty minutes, with frequent agitation. The sulphonated mixture is next diluted with its own volume of water and steam distilled. In the case of pure turpentine, a very small quantity (not more than 0.5 c.c.) of yellow oil, having a refractive index at 20° C. above 1.500, is obtained. If mineral

oil is present in the sample, it is recovered at this point, and may be identified by its characteristic odour and refractive index, which is usually below 1.400. When oil ceases to distil the steam supply is disconnected, and the mixture distilled by direct heat. As soon as oily drops appear in the distillate, hot water is added slowly to the flask from a dropping funnel, and at a slightly lower rate than the liquid is distilling, so that the temperature rises gradually. The distillation is run at about 5 or 8 c.c. per minute. In the case of adulteration with solvent naphtha, hydrolysis of the sulphonated compound begins at about 115° C.; when the temperature reaches about 170° C. excessive frothing occurs, and the distillation must be stopped. The volume of oil recovered in the distillate is multiplied by 2.2 to obtain the percentage of coal tar oil in the sample. The oil may be identified as a benzene hydrocarbon by the usual tests (nitration, etc.). The method does not give reliable results when applied to wood turpentine, whether obtained by distillation with steam or by destructive distillation, probably because the high temperatures used in the methods of manufacture break down the terpenes and resins partially, with the formation of ring hydrocarbons; if, however, more than 4 per cent. of oil is recovered from a wood turpentine, the purity of the sample is doubtful.

W. P. S.

The Gold Number of Commercial Gelatins. F. A. Elliott and S. E. Sheppard. (*J. Ind. Eng. Chem.*, 1921, 13, 699-700.)—The gold number of colloids indicates the number of mgrms. of colloid necessary just to prevent the precipitation of 10 c.c. of standard gold solution by 1 c.c. of 10 per cent. sodium chloride solution. The gold hydrosol is prepared by heating a mixture of 120 c.c. of conductivity water, 2.5 c.c. of 0.6 per cent. gold chloride solution, and 3.5 c.c. of 0.18 N-potassium carbonate solution, nearly to boiling, and adding 2 c.c. of 0.3 per cent. formaldehyde solution. Commercial gelatins differ but little as regards their gold value, and the latter does not bear any simple relation to those properties which are of chief interest to users of gelatins.

W. P. S.

INORGANIC ANALYSIS.

Phenol Red as Indicator of Acidity. A. Massink. (*Pharm. Weekblad*, 1921, 58, 1133-1136.)—For the colorimetric estimation of hydrogen-ion concentration in the region of P_H 7 to 8, neutral red is a more suitable indicator than phenol red, the latter being considerably influenced by the presence of salts. For example, in the case of a water with an electrical conductivity of $K_{18} \cdot 10^6 = 33,350$, the P_H value with neutral red was 7.10, whilst with phenol red it was 7.35. But the same water diluted 1:10, so that its conductivity became $K_{18} \cdot 10^6 = 4,250$, still gave the same P_H value (7.10) with neutral red, but only 7.05 with phenol red.

Detection of Carbon Monoxide. C. R. Hoover. (*J. Ind. Eng. Chem.*, 1921, 13, 770-772.)—A mixture of iodine pentoxide 11.0, granular pumice-stone 34.0, and fuming sulphuric acid (60 to 80 per cent. SO) 55.0 per cent. yields a green to black coloration when brought into contact with carbon monoxide. Under favourable conditions as little as 0.005 per cent. of carbon monoxide in air may be detected by

passing 500 c.c. of the air through a glass tube containing a 50 mm. by 5 mm. column of the mixture. The green colour indicating the presence of carbon monoxide disappears in a few minutes, and the tube may be used again for several successive tests. Colour standards may be prepared with pumice-stone and basic copper acetate or chromium oxide, and the test thus rendered approximately quantitative. Other unsaturated gases, such as hydrocarbons, hydrogen sulphide, hydrocyanic acid, etc., interfere, but may be removed by passing the air previously through dry active charcoal; hydrogen, methane, sulphur dioxide, nitric acid, and ammonia, are without action on the mixture.

W. P. S.

Sensitive Reaction for Copper. P. Falcicola. (*Giorn. Chim. Applic.*, 1921, 3, 354-355.)—Copper may be detected in very dilute solution by means of a solution of alkali thiocyanate mixed with a cold saturated solution of gallic acid. With this reagent a $\frac{1}{10000}$ copper sulphate solution gives a decided turbidity, whilst a $\frac{1}{100000}$ solution, if shaken with the reagent, soon develops perceptible whitish cloudiness. The cupric solution must be neutral, since the presence of mineral acid hinders the appearance of the precipitate, although the latter, after its formation, is virtually insoluble in acids; the ions Ag^+ , Pb^{++} , and Bi^{+++} , should also be absent, as they yield precipitates under the above conditions.

T. H. P.

Rapid Estimation of Copper and Iron in Aluminium Alloys.* P. Hulot. (*Bull. Soc. Chim.*, 1921, 29, 836-837.)—The alloy (2 to 5 grms.) in the form of wire, sheet, or turnings is dissolved in 20 per cent. sodium or potassium hydroxide solution, the liquid being warmed gently towards the end of the reaction and diluted with water; the iron and copper remain insoluble, and may be washed, collected, and weighed. If greater accuracy is required, the copper may be dissolved and deposited electrolytically, and iron may be dissolved and reprecipitated as hydroxide. The method avoids the filtration of gelatinous alumina, and is therefore useful for rapid work in the foundry.

R. G. P.

Estimation of Aluminium and of Aluminium Oxide in Commercial Aluminium. J. H. Capps. (*J. Ind. Eng. Chem.*, 1921, 13, 808-812.)—A weighed amount of the metal is boiled with water in a flask to expel dissolved and absorbed gases; the flask is then filled with water (previously boiled) and closed with a stopper carrying a tapped funnel, a short side tube, and a capillary delivery tube connected with a gas measuring burette. A quantity of reagent (sulphuric acid or sodium hydroxide solution) is admitted from the funnel, the side tube being opened during this operation and then closed. As hydrogen is evolved, a portion of the liquid in the flask is forced back into the funnel, a tap on the capillary being closed, thus forming a gas space in the flask. Connection with the burette is then made, the contents of the flask are heated, and, when evolution of gas ceases, the flask is filled with liquid from the funnel, and all gas driven over into the burette. The latter is provided with a water jacket and is filled with water saturated with hydrogen. If

* A similar method was described by Gemmell in the discussion on a paper by Withey (*ANALYST*, 1916, 41, 181).—EDITOR.

sulphuric acid is used to decompose the metal, allowance must be made for the quantity of hydrogen formed by the iron present in the aluminium; when sodium hydroxide is used, a similar allowance must be made for the hydrogen resulting from the action of the alkali on the silicon present. The volume of the hydrogen obtained is a measure of the metallic aluminium present in the sample; the aluminium oxide is obtained by difference.

W. P. S.

Estimation of Vanadium in Ores and Metallurgical Products. R. B. Schaal. (*J. Ind. Chem. Soc.*, 1921, 13, 698-699.)—The method depends on the reduction of vanadium pentoxide to the dioxide by prolonged boiling with hydrochloric acid, and subsequent titration of the dioxide with permanganate solution— $V_2O_5 + 2HCl = V_2O_4 + Cl_2 + H_2O$, and $5V_2O_4 + 2KMnO_4 + 6HCl = 5V_2O_5 + 2KCl + 2MnCl_2 + 3H_2O$. In a nearly neutral solution containing an excess of ammonium phosphate the end-point of the titration is sharp, and relatively large amounts of iron, chromium, molybdenum, cobalt, nickel, titanium, and zirconium do not interfere; small amounts of manganese are also without effect. In the case of steels, 2 grms. of the sample are dissolved in 50 c.c. of hydrochloric acid (1 : 1), 5 c.c. of nitric acid are added, and the solution is evaporated to 10 c.c. For ores, from 1 to 5 grms. of the sample are digested with 60 c.c. of concentrated hydrochloric acid, 5 c.c. of nitric acid and 2 c.c. of hydrofluoric acid are added, and the mixture is evaporated to dryness. With ferrovanadium, 0.5 grm. is treated with 10 c.c. of nitric acid, 2 c.c. of hydrofluoric acid are added after a time, the mixture is heated, and, when violent action has ceased, 40 c.c. of concentrated hydrochloric acid are added, and the mixture is evaporated to dryness. To the solution or residue, as the case may be, are added 40 c.c. of hydrochloric acid, the mixture evaporated to dryness, this addition of acid is repeated, and the mixture evaporated to 10 c.c. The residue is mixed with 50 c.c. of water, filtered, and the insoluble portion washed five times with cold water, 15 c.c. being used each time. The filtrate is treated with 60 c.c. of 15 per cent. ammonium phosphate solution, ammonia is added until a precipitate forms, and this is dissolved by the addition of a slight excess of hydrochloric acid. The solution is then diluted to 300 c.c., and titrated with standardised potassium permanganate solution.

W. P. S.

Electrometric Estimation of Bromate, Dichromate, Nitrite, and Chloride Ions. W. S. Hendrixson. (*J. Amer. Chem. Soc.*, 1921, 43, 1309-1317.)—Methods are described for the direct electrometric titration of iodide with bromate and dichromate, and the indirect titration of bromate with permanganate. The direct titration of iodide can be made in 2 *N*-sulphuric acid solution, but much time is required to reach the true end-points, and the indirect titration, finishing with permanganate, is preferred. For the electrometric estimation of nitrite, a weighed quantity of standard permanganate solution is placed in the titration vessel, diluted, and 10 *N*-sulphuric acid is added in amount sufficient to make the final solution about 1.5 *N*. The mechanical stirrer is started, and the nitrite solution added slowly in such quantity that 5 to 10 per cent. of the permanganate remains unchanged. After five minutes an excess of standard iodide solution is added, and the titration completed with permanganate solution.

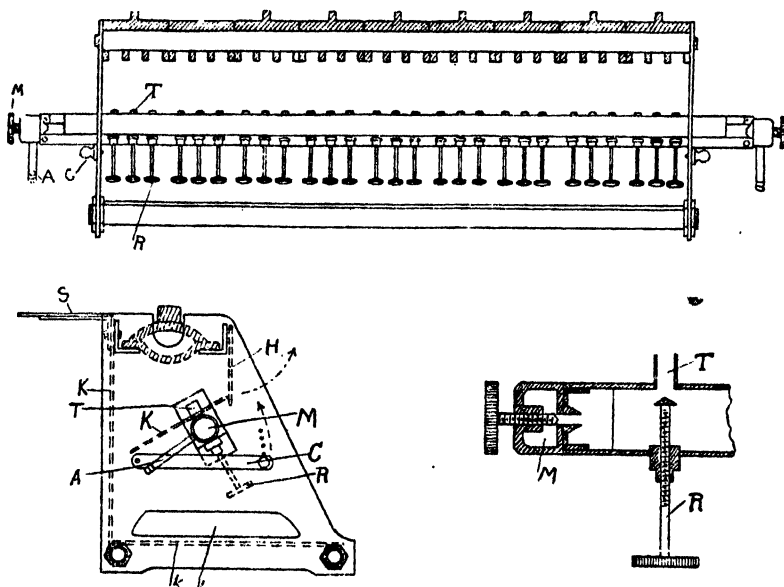
W. P. S.

Estimation of Sodium Hyposulphite. J. H. Smith. (*J. Amer. Chem. Soc.*, 1921, 43, 1807-1808.)—About 0.4 grm. of the solid sodium hyposulphite (usually, but wrongly, termed sodium hydrosulphite) is placed in a beaker, and treated with a large (100 per cent.) excess of ammoniacal silver nitrate solution; the mixture must not be warmed. As the salt dissolves it is oxidised, and metallic silver is precipitated. The precipitate is collected on a Gooch crucible, washed with ammoniacal ammonium nitrate solution, the crucible then placed in the same beaker, and the silver dissolved in warm nitric acid. The solution is boiled to expel nitrous acid, the crucible is removed, and the solution is diluted and titrated with $\frac{N}{10}$ potassium thiocyanate solution, ferric alum being used as indicator. The oxidation proceeds according to the equation: $\text{Na}_2\text{S}_2\text{O}_4 + 2\text{AgNO}_3 + 4\text{NH}_4\text{OH} = 2(\text{NH}_4)_2\text{SO}_4 + 2\text{NaNO}_3 + 2\text{H}_2\text{O} + \text{Ag}_2$; one molecule of sodium hyposulphite is, therefore, equivalent to two molecules of potassium thiocyanate.

W. P. S.

PHYSICAL METHODS, APPARATUS, ETC.

Improved Gas Combustion Furnace for Use in Organic Analysis. T. J. Hedley. (*J. Chem. Soc.*, 1921, 119, 1242-1243.)—A light steel framework similar to that employed in the usual form of furnace, but covered at the back and base with uralite, and fitted with a horizontal door of the same material in front, carries a row of trough tiles and covers at the top. The multiple burner consists of a large metal tube, closed in the centre, and fitted at each end with a gas



inlet and a screw valve. The separate burners fitted on one side of this tube are controlled by screw cone valves projecting from the opposite side of the tube. The whole burner is supported by spring arms fixed upon the end plates of the furnace, and the burners can be adjusted at different distances from the tiles. The construc-

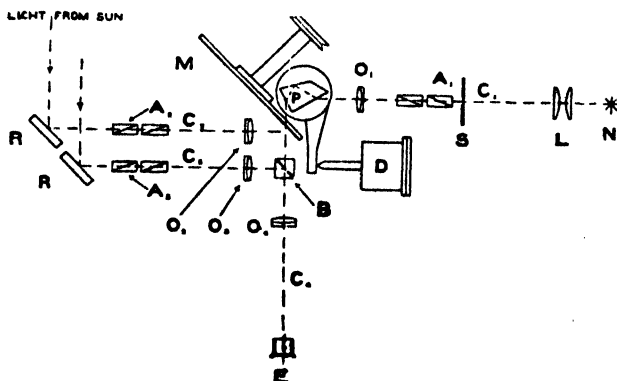
tion is shown in the diagram: *A*, gas inlet; *C*, spring arms; *H*, hinged uralite screen; *K*, fixed uralite screens; *M*, main gas regulator; *R*, cone valves; *S*, hot tile shelf; *T*, burner tubes. The advantages claimed for this type of furnace are: Portability, the weight being less than 30 pounds; small gas consumption; protection from excessive radiation; rapid control of temperature by adjustment of the gas; and economy in cost of construction.

T. J. W.

Rapid Method of Determining the Density of Air. G. P. Baxter. (*J. Amer. Chem. Soc.*, 1921, 43, 1817-1818.)—A sealed globe of known exterior volume is weighed under accurately known conditions of temperature, pressure, and humidity; from these conditions the density of the atmosphere at the time of weighing and the buoyant effect of the air on the globe and weights are computed. By adding the difference between the buoyant effect on the globe and on the weights to the weight of the globe in air, the weight of the globe *in vacuo* is found. If the globe be weighed in the air under any conditions, the weight of the air displaced by the globe in excess of that displaced by the weights is found by subtraction from the weight *in vacuo*; this difference, divided by the volume of the globe, less the volume of the weights, gives the atmospheric density.

W. P. S.

Measurement of Colour. C. E. K. Mees. (*J. Ind. Eng. Chem.*, 1921, 13, 729-731.)—The most convenient and satisfactory colorimeter for reading the colour of an object in absolute units—namely, the wave length of the dominant hue and the percentage—is the monochromatic analyser. In this instrument the colour to be measured is balanced by means of a mixture of spectrum colour and white light. For instance, to measure the colour of a green tinted paper, the paper is placed so that the light reflected from it falls into the colorimeter (see illustration). In the photometric field at *B* is seen one part of the field filled by the green light from the coloured paper, whilst the other part of the field can be illuminated by means of a spectrum, to which can be added any proportion of white light that is required. The



wave length of the spectrum light is changed by means of the drum until the tint appears to be approximately right; white light is then added from the beam *C*, reflected from the glass surface *M*, until the two tints are about the same, the intensity

of the two fields being kept equal to each other. The tint may now require a little correction, and when this is almost exactly right the admixed white light may require another correction. By this method the colour may be matched rapidly, and the dominant tint is then read off on the spectrum drum, and the percentage of admixed white light is measured photometrically in the instrument. A red may be specified as being of dominant tint wave length 650, corresponding in tint with the red hydrogen line; a yellow might be of dominant tint 580; a greenish-yellow would be 570, and a yellowish-green 560.

W. P. S.

Repair of Crystallised Glass Apparatus. H. Bailey. (*J. Amer. Chem. Soc.*, 1921, 43, 1319.)—The method consists in holding the nearly-melted glass in a sodium flame for a few seconds. A piece of Pyrex tubing wrapped with a few layers of asbestos paper saturated with sodium chloride solution is held in the blow-pipe flame in such a position that the sodium vapours envelope the devitrified glass; all signs of crystallisation usually disappear by the time the glass is soft enough to work. (*Of. ANALYST*, 1921, 164.)

W. P. S.



SAFEGUARDING OF INDUSTRIES ACT, 1921.

THE LIST OF ARTICLES CHARGEABLE WITH DUTY UNDER PART I. OF THE ACT.

IN pursuance of Sec. 1 (5) of the Act, the Board of Trade have prepared a list defining the articles which are to be taken as falling under the following general description set out in the Schedule—viz.: Optical glass and optical elements, whether finished or not (List A); optical instruments (List B); scientific glass ware (List C); laboratory porcelain (List D); scientific instruments (List E); gauges and measuring instruments of precision of the type used in engineering machine shops (List F); compounds (not including ores or minerals) of thorium, cerium, and other rare earth metals (List G); synthetic organic chemicals (other than synthetic organic dyestuffs, colours, and colouring matters imported for use as such, and organic intermediate products imported for their manufacture), analytical reagents, all other fine chemicals (except sulphate of quinine of vegetable origin), and chemicals manufactured by fermentation processes (List H).

Articles falling under any of the general descriptions in paragraph 3, or included in the Lists A to H, will be chargeable with duty under Part I. of the Act on and after October 1, 1921.

According to Sec. 1 (4) of the Act, no duty shall be charged under this section in respect of a compound article containing an article subject to duty if the compound is of such a nature that the article liable to duty has lost its identity, and any dispute on this point shall be determined in like manner as disputes as to whether goods are goods specified in the Schedule to the Act.

Sec. 1 (5) provides that if within three months after the publication of any list of articles according to Sec. 1 of the Act any person appearing to the Board to be interested delivers to the Board a written notice complaining that any article has been improperly included in or excluded from the list, the Board shall refer the complaint to the arbitration of a referee to be appointed by the Lord Chancellor,

who shall not be an official of any Government department, and the decision of the referee shall be final and conclusive, and the list shall be amended so far as is necessary in order to give effect to the decision, without prejudice, however, to the validity of anything previously done thereunder.

LIST A—OPTICAL GLASS AND OPTICAL ELEMENTS.—Lenses, photographic (unmounted); lenses, spectacle (mounted or unmounted).

LIST B—OPTICAL INSTRUMENTS.—Binoculars, prismatic; field glasses; microscopes, with or without lenses; opera glasses; ophthalmometers; photographic cameras, with or without lenses; sight-testing apparatus; oculists' trial sets and cases.

LIST C—SCIENTIFIC GLASS-WARE.—Ampoules; barometers, mercury, in glass; beakers; bottles, culture, weighing, and Wolff's; funnels, separating, thistle, and filtering; flasks, various; jars, various; Kipp's apparatus; lecture and experimental apparatus; mortars and pestles; percolators; pipettes; pycnometers; retorts (laboratory); syringes, glass; specific gravity apparatus; thermometers; tubes.

LIST D—LABORATORY PORCELAIN.—Baths, air and water, and cover plates; burners, Bunsen; centrifuges; crucibles; dishes, evaporating; filter funnels; funnels, plain and filtration, including Buchner's funnels; retorts; rings; spatulas; spoons; tubes, combustion; trays; water baths.

LIST E—SCIENTIFIC INSTRUMENTS.—Autoclaves, laboratory; balances, analytical, assay, chemical, and other precision; hydrometers; hygrometers; influence machines (electric) and other static machines; microtomes; physical lecture and experimental apparatus.

LIST G—COMPOUNDS OF CERIUM, THORIUM, AND OTHER RARE EARTH METALS.—The list includes 72 compounds of thorium, 68 of cerium, and 16 of other rare earth metals.

LIST H—ANALYTICAL REAGENTS, FINE CHEMICALS, ETC.—The list includes over 4,000 articles. Those to which the letter D is attached are chemicals covered by the Dyestuffs (Import Regulation) Act, 1920, and come within the scope of the present Act only when importation has been permitted for purposes other than those with which the Dyestuffs Act is concerned.

The letter R signifies that the chemical is included only when it is "pure," "puriss.," "extra pure," "B.P.," "Ph.G.," "A.R.," "for analysis," "reagent," or when it is of special quality for meeting special tests of purity, and not when it is the crude product.

The analytical reagents specifically named include, *inter alia*, the following: Acetanilide (D); acetic anhydride, acetone (R); Acid, acetic (80 per cent. grade or higher); arsenious (R), benzoic (D), boric, carbolic (synthetic) (D); carbolic (R); chromic, citric, formic, gallic, hydriodic, hydrobromic, hydrochloric (R), hydrocyanic, hypophosphorous, iodic, lactic, phosphoric, phosphorous, picric (D), pyrogallie (D), salicylic (D), sulphuric (R); tannic (R); tartaric. Alcohol, absolute; amyl (R), methyl (R). Alum, ammonia (R); chrome; potash (R); ammonium nitrate (R) and 115 compounds of ammonium; aniline (D); antimony chloride (R); barium carbonate (R) and chloride (R), and 75 other barium compounds; bismuth, 64 compounds; bromine; cadmium salts; calcium carbonate (R), phosphate (R), sulphate (R), and 95 other compounds; carbon disulphide (R); carbon tetrachloride; cellulose acetate; chloroform; copper sulphate (R), and 68 copper compounds; cresols; exalgine (D); formaldehyde; guaiacol compounds; hæmatoxylin, heliotropine, iodine (R); iodoform; iron (R), iron perchloride (R), iron sulphate (R), and 80 other iron compounds; lactose (R); lead carbonate (R), oxide (R), subacetate (R), and 54 other lead compounds; lithium, bromide, and 47 other lithium compounds; magnesium sulphate, and 77 other compounds; maltose (R); mercury chloride, bichloride, and 54 other mercury compounds; nicotine (R); perhydrol, phenazone; phenol (D); phenolphthalein; potassium bichromate (R), carbonate (R), hydroxide (R), ferricyanide; ferrocyanide (R), and 145 other salts; quinine, 76 salts; resorcinol; Rochelle salt; saccharin;

salicin; silver nitrate, oxide, and 48 other salts; sodium bicarbonate (R), hydroxide (R), hyposulphite (R), sulphate (R), sulphite (R), thiosulphate (R), and 225 other salts; strontium bromide, and 34 other strontium salts; strychnine, 23 compounds; thallium acetate; thymol, tin oxide, and other salts; uranium nitrate, veronal, xylene, zinc carbonate (R), chloride (R), oxide (R), sulphate (R), and 71 other zinc salts.

Among the alkaloids in the list are: Epinephrine, ergamine, ergotoxine, novocaine, pellitterine, scopolamine, solanine, and yohimbine, and their salts.

Copies of the list (price 9d.) may be obtained from H.M. Stationery Office at the following addresses: Imperial House, Kingsway, W.C. 2; 28, Abingdon Street, S.W. 1; 37, Peter Street, Manchester; 1, St. Andrew's Crescent, Cardiff; 23, Forth Street, Edinburgh; or from E. Ponsonby, 116, Grafton Street, Dublin.



NEW CHEMICAL STANDARDS.

(ANALYTICALLY STANDARDISED SAMPLES.)

ANOTHER new plain carbon steel is now ready for issue—viz., "R," which fills the vacancy in the series for a carbon standard between "I" (carbon 0.521 per cent.) and "SI" (carbon 0.921 per cent.). It is also intended to serve as a high manganese standard and a sulphur standard in high carbon steel.

The analyses have been undertaken as usual by a number of experienced chemists representing the following interests: British Government Department; U.S. Bureau of Standards; Referee Analysts, independent; Railway Analysts, representing users issuing specifications; Works Analysts, representing makers and users.

The standard figures are as follows—CARBON STEEL "R": Carbon (combustion), 0.786; carbon (colorimetric), 0.790; silicon, 0.22*; sulphur, total by oxidation, 0.053; sulphur, evolved as sulphide, 0.50; phosphorus, 0.058*; manganese, 0.914; arsenic, 0.03*; copper, 0.02*; Ni, Cr, W, Mo, none detected by qualitative tests.

The standards may be obtained in 500, 100, or 50 gm. bottles, either direct from Organising Headquarters, 3, Wilson Street, Middlesbrough, or through any of the leading laboratory furnishers, at a price just sufficient to cover the cost. A certificate, giving the names of the analysts co-operating, the types of methods used, and a detailed list of the results, will be supplied with each bottle.

REVIEWS.

AIDS TO CHEMISTRY. By WILLIAM PARTRIDGE, F.I.C. Pp. viii + 280. London: Baillière, Tindall and Cox. 1921. Price 6s. net.

That our present system of examinations is far from ideal is almost a commonplace, though no alternative method of deciding a person's qualifications has, so far, met with general approval. There are many, both teachers and pupils, to whom the passing of examinations is wellnigh the sole aim of study. From this state of affairs it arises that a class of books has come into existence whose only object seems to be just to push the student through his next examination in the shortest possible space of time. Such books are, as a rule, soulless collections of information, which, though doubtless answering this purpose, can hardly be expected to inspire the

* Approximate.

learner with enthusiasm or to provide him with a real scientific education. Unable to stand the strain of the unromantic "grind" necessary to acquire the requisite knowledge of detail, his energies begin to flag, and, with a sigh of relief, he drops the unwelcome subject as soon as the examination has been "successfully passed."

The small volume under consideration is somewhat of this type, attempting to cover almost the whole range of chemistry in less than 300 pages. Roughly speaking, 50 pages are allotted to general theory, 50 to the non-metals, and another 50 to the metals; after a short general account of the special features of organic chemistry, aliphatic compounds receive about 70, and aromatic compounds 15 pages; there are also shorter sections dealing with cyanogen and heterocyclic compounds. The book, on the whole, is clearly written, and a large amount of information is condensed into a small compass. The author's choice of material is decidedly open to question, and the volume would be improved by devoting increased space to the more important portions of the subject, omitting reference to various compounds of little or no value to the medical and pharmaceutical students for whom the book is intended.

As a kind of summary for revision purposes, this manual will no doubt be of value, but as a textbook for general study it is not to be recommended.

A. F. KITCHING.

ANALYSES AND ENERGY VALUES OF FOODS. By R. H. A. PLIMMER, D.Sc. Pp 255. London: H.M. Stationery Office. 1921. Price 6s. net.

The well-known work of König was for many years practically the only available storehouse of analytical data as to the composition of foods, and much of its information referred to products of Continental origin. The series of analyses made by Atwater and Bryant under the auspices of the U.S. Department of Agriculture was remarkable for its completeness, and when published in 1906 in tabulated form soon gained an unchallenged position as a standard work of reference on the composition and energy values of American foods. Hitherto, however, there has been no similar compilation of systematic analyses of British food products, and this little handbook, therefore, fills a distinct gap.

The investigation was primarily undertaken to obtain trustworthy figures which could be used as a guide by the army medical authorities in supplying the army with the right food under the conditions of rationing prevailing during the war, but it is so full of valuable detail, much of which is new, that it should serve as the foundation upon which definite standards for food values might be based.

The first impression on taking up the book is of the immense amount of work which it embodies, and Dr. Plimmer may be congratulated upon successfully carrying through, almost single-handed, this series of some 900 analyses, involving, as he tells us, over 20,000 weighings.

The work was not intended to supersede the results of Atwater and Bryant, but rather to supplement them and to provide average data for each group of foods.

An outline of the methods of analysis used is given in a short introduction, and the method of calculating the energy values is explained. From the point of view of the chemist, for whom, however, the book is not primarily intended, a little more discussion on the question of the energy value would have been an advantage. It is

not pointed out, for instance, that the calorific values calculated from the composition agree fairly closely with the values actually determined by combustion in the case of certain foods such as meat, milk, white bread, potatoes, and fruits, whereas in other cases, notably peas and many other vegetables, the theoretical values fall far below the actual values. It is only by a compensation of the inherent errors that the calculated and actual values usually agree fairly well when a mixed diet is under examination.

The question of the influence of the digestibility factor is mentioned, but not discussed at any length, although obviously it must have great weight, especially in the case of vegetable products.

Still, whatever method of calculating energy values be ultimately adopted, all the factors used in calculating the proximate composition of the different foods are given, so that it will be possible to recalculate the data upon any other basis.

The tables include not only analyses of all the more common kinds of food, but also, wherever necessary, show the component parts. For example, the average percentages of food products, waste, and bone, in an ox are given, and in the case of the separate joints the fat and skin and lean meat, and analyses of these separate portions are given.

In connection with the analyses of war bread, it is interesting to note that the main difference from pre-war bread, analyses of which by Cribb and Elliot are quoted in full, was in the appearance, the composition being very similar. Parenthetically it might be suggested that this is one of the cases where the digestibility factor might have been taken into consideration.

A useful summary of the food containing the accessory factors, the vitamins A, B, and C, is given in an appendix, so that it can be seen at once whether a particular food is rich or poor in any of these active principles.

In short, as a work of reference this little book is one which every chemist engaged in the examination of foods should have and keep in constant use.

EDITOR.

ANIMAL PROTEINS. By H. G. BENNETT, M.Sc. Pp. xii + 287. London: Baillière, Tindall and Cox. 1921. Price 15s. net.

The title of this book is misleading. It contains a well-written account of the methods employed in the tanning industry, together with a description of the manufacture of gelatin and glue. In this way the industrial chemistry of collagen and gelatin occupy 268 pages, while all other animal proteins are relegated to 14 pages at the end of the book, in which the matter is so condensed as to be practically without value. We are told that the object of the book is to give a general survey of the industries involved, and as far as the leather, gelatin, and glue trades are concerned, it succeeds very well in its purpose. To the young chemist wishing to enter one of these industries the book will be of great value, though he must remember that technical procedure varies so greatly from firm to firm that no account of the details of manufacture can be of universal application. The statement on p. 164, for instance, that "after puering (chrome goat and sheep) it is common to give a low temperature drench" is one which is open to challenge. This, however, is a minor point, detracting little from the value of the whole.

It is with the attempted theoretical explanations of the various stages in leather manufacture that there is more room for criticism. For instance, in the section on "the preparation of pelt" the question as to what actual processes are going on in the lime-pits is taken up at length. One practical result of liming is that the skins can be unhaired at the end of the process. The theoretical question is, How is this brought about? We find the following statements: p. 20, "The function of lime is complex. . . . Its main purpose, however, is that of a partial antiseptic"; on the same page, "all tannery lime liquors are swarming with putrefactive bacteria"; p. 21, "the excess of undissolved lime has an accelerating effect on bacterial activity"; and "the depilating organisms of liquors are probably mostly anaerobes"; "lime plays an essential part in assisting the putrefactive fermentation." Mr. Bennett's theory is evidently that unhairing is brought about by bacterial activity in the lime-pits. Now, although it is true that a number of different species of organisms have been isolated from tanyard lime liquors, it remains to be proved that these bacteria are in a state of functional activity while in the limes. Lime liquors have a hydrogen-ion concentration of $P_H = 12.4$, and no evidence has yet been brought forward to show that the number of bacteria increases in the pit during the process of liming. It is now known that many so-called antiseptics—*e.g.*, toluene, ether—act not by killing bacteria, but by inhibiting their activity. The mere isolation of bacteria from a fluid is not proof that the bacteria were playing any active rôle in any chemical processes going on in it. Bacterial activity in the lime-pits has always been assumed, but never proved. In view of the reaction the omission is serious.

Mr. Bennett's theory of the influence of electrolytes on gelatin gels would, we fear, increase rather than decrease the difficulties of any leather chemist anxious to learn the background of his subject. "Lyotrope influence" and "adsorption" are freely invoked as explanations, and in one place (p. 217) we are told "it is sometimes a difficult problem to decide whether an increase or decrease in swelling is due to lyotrope or adsorptive influence." With this statement we can cordially agree. Mr. Bennett's arguments are ingenious, and his theories have doubtless been a guide to him in his own work; but we feel that for the general scientific public, as also for leather chemists, Professor Procter's work and theories will continue to hold the pre-eminent position. It is becoming clearer every day that "adsorption" is a term which describes, but does not explain, concentrations at interfaces, and "lyotrope influence" is a mystery which has happily been dispelled by the recent work of Loeb. Mr. Bennett considers that swelling is due to the repulsion of similarly charged colloidal ions, and refers constantly to gelatin as "primarily a positive colloid" (pp. 45, 213, etc.). What he means by "primarily" is difficult to make out. Gelatin is a positive colloid in acid solutions, a negative colloid in alkaline ones, and neutral at $P_H = 4.6$. The nature of the charge, therefore, is not inherent in the gelatin, but depends on the reaction of the surrounding medium. He also states that "pelt is a gel." This seems a somewhat archaic use of the term "gel," and one which is to be avoided.

Mr. Bennett's book, regarded as a treatise on the manufacture of leather, fills an obvious gap in scientific industrial literature, and as such is to be thoroughly commended.

DOROTHY JORDAN LLOYD.



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THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

AN Ordinary Meeting of the Society was held on Wednesday, November 2, 1921, in the Chemical Society's Rooms, Burlington House. The President, Mr. Alfred Smetham, was in the Chair.

Certificates were read for the first time in favour of Messrs. Jack Cecil Drummond, D.Sc. (Lond.), F.I.C., Alfred William Long, John Charles Mellersh, and Charles Harold Wright, M.A. (Cantab.), F.I.C.

The following were elected Members of the Society: Messrs. James Frederick Fothergill Rowland, B.A. (Cantab.), A.I.C., and William Norman Stokoe, B.Sc. (Lond.), A.I.C.

The following papers were read: "An Improved Specific Gravity Apparatus," by C. Butler Savory, M.D., M.Ch., and "The Testing of Foodstuffs for Vitamins," by J. C. Drummond, D.Sc., F.I.C., and A. F. Watson, D.Sc., A.I.C.

* * * * *

MORPHINE, CODEINE, AND NARCOTINE IN INDIAN OPIUM.

By JITENDRA NATH RAKSHIT.

THE chemistry of Indian opium naturally differs somewhat from that of Turkish opium on account of variation in soil, climate, seeds, method of cultivation, of lancing the poppy capsules, and in the collection of opium. It would be beyond the scope of the subject to dwell more fully on the question of how the constituents of opium vary with the variations in the above conditions. It has been found, however, in the case of crops of poppy grown by Mr. G. O. B. Power, of the Government Opium Department, that healthy plants, at the first lancements of their capsules, yield opium with higher morphine content than that from weaker plants, or than the drug otherwise collected by subsequent lancements of the same capsules.

MORPHINE.—The author has been estimating morphine in no fewer than 3,000 samples of opium a year by the British Pharmacopœia and by the polarimetric methods (Rakshit, ANALYST, 1918, 43, 321), but has never met with a sample of opium which could justify the remark in "Allen's Commercial Organic Analysis" (Vol. VI., fourth edition, p. 408): "East Indian opium is, as a rule, remarkably

weak in morphine, the proportion being sometimes as low as 2·5 per cent., more commonly 3·5 and 5, and occasionally as high as 8 or 9 per cent." During this last half century there has been no such change as would produce any improvement in Indian opium in respect of its morphine content. As a matter of fact, not a single bag of opium has been received from the districts during the last seven years which gave less than 7 per cent. of morphine. The bulk of them gave 8·5 to 10·5 per cent., whilst a good quantity showed 10·5 to 12·5 per cent. Obviously, then, the former analysts must have adopted grossly erroneous methods to obtain such low results.

The British Pharmacopœia process has been well criticised by quite a number of authors, but the way in which results obtained by this process are vitiated by the tropical climate of India has not been noted to any extent. In Upper India the humidity during the summer months becomes often as low as 4 and 5, and under such conditions evaporation during the trituration in the mortar and the filtration is quite rapid, and effectively concentrates the solution. The filtered lime solution of the opium often does not amount to 51 c.c. in the case of Indian opium, as required by the B.P. process; whilst when the ether is transferred to the filter-papers, a part of it rapidly evaporates during filtration, leaving a yellowish resinous deposit of alkaloids on the top portion of the upper filter-paper, and seldom any on the counter-poised lower one. The 10 c.c. of ether recommended in the process for washing the filter-paper does not completely wash this deposit, nor does it act uniformly on the two papers. In addition to this, a small quantity of ether left on the filter-paper, after the aqueous mother liquor has passed through, always leaves a residue on the upper filter-paper only. The deposit left on the evaporation of the ether is never removed by the recommended subsequent washing with morphinated water. Such residues, however, are always found to be soluble in ether or acid.

In some laboratories methods of the United States Pharmacopœia are adopted, and since these are purely gravimetric methods, the errors due to the evaporation during the preparation of the solution and the filtration are not introduced. The prescribed washings with alcohol and ether completely remove the residue left on evaporation of the ether which escapes filtration. It must not be assumed, however, that this method is free from defects. The substance dissolved by the lime water has been taken to be pure morphine, which, however, is not the case, as is shown below. Moreover, no correction is made for the retention of morphine by the mother-liquor.

Several experiments have been made to compare the results obtained by the polarimetric process with those of the U.S.P. process, and a few typical results are given below :

Sample.		Polarimeter Result.		Morphine Strength, U.S.P.	
		Per Cent.		Per Cent.	
Ghazipur	...	10·2	...	9·8	...
Lucknow	...	10·1	...	9·7	...
Cawnpore	...	9·8	...	9·6	...

To ascertain the amount of morphine left in solution after the precipitation of the base in the U.S.P. process, the aqueous mother liquor was thrice extracted with

ether, 100 c.c. being used each time to get rid of any codeine which might still be present in solution, evaporated to a small bulk, acidified with 1 c.c. of hydrochloric acid, made up to 100 c.c., decolorised with 20 grms. of animal charcoal, and polarised in a 200 mm. tube.

Sample.	Reading on Ventzke Scale.	Total Mor- phine in 100 c.c. gram.
1 gram. of morphine hydrochloride (dry) instead of extract from 10 grms. of opium	... -1.3 ...	0.18
Karmansa	... -1.7 ...	0.23
Ghazipur	... -1.8 ...	0.24
Gwalior	... -2.6 ...	0.36

A quantity of 0.5 gram. of morphine, obtained by the U.S.P. process, was titrated with $\frac{N}{10}$ sulphuric acid, with litmus as indicator, and the neutral or slightly acid solution was made up to 50 c.c., filtered, and polarised in a 200 mm. tube. The specific rotation was calculated by assuming the gross weight to be entirely due to morphine, and by regarding as morphine everything which neutralised acid.

Sample.	Morphine as Obtained by Titration.	Reading on Ventzke Scale.	Specific Rotation based on—	
			Gross Weight.	Titration Value.
Ghazipur	Per Cent. 98.34	-6.72	-116.5°	-119.5°
"	97.34	-6.80	-118.5°	-121.2°
"	96.80	-6.72	-116.5°	-120.2°
Pure morphine	—	—	-127.5°	-127.5°

Morphine (0.5 gram.), obtained by the U.S.P. process, was shaken with 50 c.c. of freshly made lime water in a 250 c.c. Erlenmeyer flask, the liquid filtered through counterpoised double filter-papers, and the residue washed with lime water until the filtrate, on acidification, ceased to give any precipitate with Mayer's reagent. The substances dissolved and undissolved by the lime water were usually determined gravimetrically. The lime-water solution was made up to 250 c.c. with lime water, and polarised in a 200 mm. tube, and the specific rotation was calculated by taking the weight of morphine to be that of the substances dissolved by lime water. The following results, however, show that the substances dissolved by the lime water are not all morphine:

Sample.	Morphine as Obtained by Solution in Lime Water.	Reading on Ventzke Scale.	Specific Rotation of Sub- stance Accepted as Morphine.
Pure morphine	Per Cent. 100.0	-1.4°	-60.68°
Cawnpore opium	92.4	-1.2°	-56.3°
Ballia opium	95.8	-1.2°	-53.5°

CODEINE.—Next in importance to morphine among the opium alkaloids is codeine; so its study requires some consideration. Since only very unsatisfactory determinations of its solubility were available, a fresh series of determinations has been made with all the solvents generally used for practical purposes. An excess of pure, well-powdered, dry codeine was shaken in conical flasks, at intervals, during two hours with the different solvents at different temperatures (varying not more than 2° C.), and 20 c.c. of the clear solution then withdrawn by means of a hot pipette.

SOLUBILITY OF CODEINE IN GRMS. PER 100 C.C. OF SOLUTION AT
DIFFERENT TEMPERATURES.

Solvents.				20° C.	30° C.	40° C.	50° C.	60° C.	70° C.	80° C.	90° C.	100° C.
Ether	...	...	...	2.5	2.8	—	—	—	—	—	—	—
Acetone	...	...	...	16.3	18.0	21.1	29.1	—	—	—	—	—
Chloroform	...	...	...	34.6	52.3	53.8	72.1	—	—	—	—	—
Benzene	...	...	...	7.3	9.3	15.5	23.4	38.4	58.8	63.8	—	—
Toluene	...	...	...	5.5	6.8	10.4	19.9	32.4	44.8	56.4	73.6	—
Alcohol	90	per cent.	V/V	...	22.2	34.3	50.0	—	—	—	—	—
"	60	"	"	...	20.4	48.6	59.4	65.9	—	77.0	—	84.1
"	50	"	"	...	20.0	41.9	50.6	64.7	—	75.0	—	81.4
"	40	"	"	...	19.5	26.4	40.0	51.5	—	64.0	—	78.8
"	30	"	"	...	7.8	11.6	21.6	36.0	47.1	49.2	63.7	77.4
"	20	"	"	...	3.0	3.8	7.0	11.1	18.0	26.4	47.7	61.4
"	10	"	"	...	1.5	1.8	2.4	3.1	4.1	5.2	9.9	15.8
Ammonia	10	"	W/V	...	0.9	1.2	1.5	1.8	2.3	2.3	3.2	—
"	5	"	"	...	1.1	1.2	1.4	1.7	2.0	2.1	2.6	—
"	1	"	"	...	1.0	1.2	1.5	1.8	2.0	2.1	2.3	2.6
Water	...	...	...	...	0.9	1.0	1.1	1.3	1.5	1.8	1.9	2.4

For the estimation of codeine in opium the method described by Andrews (ANALYST, 1911, 36, 489) has often been adopted. It is not only very lengthy, but has also several defects, more particularly when applied to Indian opium. The resinous mass which separates on the addition of sodium salicylate solution has been said to contain thebaine, but, in reality, is practically free from any alkaloid, and consists chiefly of salicylic acid and colouring matter. The results thus obtainable are hardly concordant, and the codeine isolated is seldom free from other alkaloids; the latter fact was ascertained by polarimetric examination. Codeine obtained by this process was titrated with $\frac{N}{10}$ sulphuric acid, the solution made up to 50 c.c. and polarised, and the specific rotation calculated upon the corrected weight obtained from the titration.

The figures clearly show that the codeine obtained by titration does not represent either the actual amount of codeine present or that contained in opium. Annett and Sen (ANALYST, 1920, 45, 321) have described a method for the extraction of codeine with toluene from the lime solution of opium. Attempts have been made to estimate codeine by this process in the case of twenty samples from different places, thoroughly representative of the total poppy cultivation in India, but unfortunately

in every case, the toluene emulsion could not be broken up, and hence the estimations were found to be impracticable. Moreover, the authors appear to have taken no precaution to remove porphyroxine, which is always extracted to some extent by almost all immiscible solvents from the lime solution of opium and in a moderate degree by toluene (Rakshit, *J. Chem. Soc.*, 1919, 457).

Sample.	Weight of Codeine obtained—		Specific Rotation.
	Gross.	Corrected by Titration.	
	Grm.	Grm.	
Codeine, pure	0.200	0.200	-137.5°
Ghazipur opium	0.200	0.167	-124.8°
Lucknow "	0.180	0.142	-130.0°
Gwalior "	0.108	0.100	-130.0°
Simla "	0.104	0.88	-132.0°
Cawnpore "	0.089	0.068	-128.0°
Azamgarh "	0.160	0.120	-128.0°

A method for the estimation of codeine has been worked out. The opium alkaloids more or less soluble in ammonia solution which require consideration are: Morphine, pseudo-morphine, thebaine, meconidine, codamine, laudanine, laudanidine, protopine, narceine, and porphyroxine, whilst the rest may be disregarded, being thrown out of solution by ammonia. Morphine, pseudo-morphine, laudanine, and narceine, are practically insoluble in ether, and the others—viz., thebaine, meconidine, codamine, laudanine, protopine, and porphyroxine—are much less soluble in ether than codeine; but, with the exception of porphyroxine, these are only nominally present in Indian opium. Hence, the alkaloid last mentioned is the only one which would cause much difficulty if an attempt were made to isolate codeine by a method based on the above differences in properties.

An aqueous extract of opium was treated with strong ammonia (sp. gr. 0.880) and filtered, the filtrate shaken with three successive portions of ether to eliminate narcotine, the ethereal extract shaken with a 1 per cent. solution of acetic acid, and the acid solution of alkaloids separated and neutralised with calcium carbonate. When the alkaloid was re-extracted with ether by making the filtered solution alkaline with sodium hydroxide the residue, left on evaporation of the ether, did not crystallise like codeine, and, on warming with a little dilute hydrochloric acid, gave the usual red coloration of porphyroxine, which was thus proved not to have been eliminated by this process.

It was then observed that when porphyroxine was heated with dilute hydrochloric acid on a steam bath it was rapidly changed into a red colouring matter, which, when treated with caustic alkalis, gave a precipitate insoluble in ether. Codeine, under such conditions, did not undergo any change. After numerous trial experiments with this process of eliminating porphyroxine from codeine, the following scheme was ultimately found to give satisfactory results:

Twenty grms. of powdered opium and 200 c.c. of water are shaken for three

hours or more in an Erlenmeyer flask and filtered, and 100 c.c. of the filtrate are added to 20 c.c. of strong ammonia solution contained in a similar conical flask, and the mixture shaken for an hour and then filtered. One hundred c.c. of this filtrate are thrice extracted with ether in a 500 c.c. stoppered separator, 100 c.c. being used each time. The ethereal extracts are filtered into another 500 c.c. separator, and the filter-paper rinsed with 20 c.c. of ether. The extract and washings are twice shaken for ten minutes with a 10 per cent. W/V solution of hydrochloric acid, 25 c.c. being used at a time. The two acid extracts are evaporated to dryness in a basin on the steam bath. The residue thus obtained, which is generally of a dark pink colour, is dissolved in 30 c.c. of water, slightly warmed on the steam bath, the solution filtered if necessary and transferred to a separator, 50 c.c. of ether and 10 c.c. of a 10 per cent. solution of pure sodium hydroxide added, and the mixture shaken for ten minutes. The aqueous layer is transferred to another separator, and the extraction repeated twice more with similar quantities of ether. The ethereal extracts are dried over two or three lumps of calcium chloride and filtered, the separator and the filter-paper washed with 20 c.c. of ether, the filtrate and washings evaporated to dryness, the residue dissolved in 10 c.c. of $\frac{N}{10}$ sulphuric acid, and the solution titrated back with $\frac{N}{10}$ alkali with litmus indicator. The codeine present is calculated from the results, or, if suitable, the acid solution is filtered, made up to 50 c.c., and polarised in a 200 mm. tube:

$$\text{Per cent. of codeine in opium} = \frac{\text{Ventzke reading} \times 100 \times 0.3468 \times 1.2 \times 10}{- 137.5 \times 2 \times 2}$$

The following are the results of the estimation of codeine in some typical samples by the above process:

Sample.				Codeine.	
1.	Two grms. of codeine (instead of 20 grms. of opium)			99.6	per cent.
2.	Ghazipur opium	...	...	1.63	"
3.	" (duplicate)	...	...	1.84	"
4.	Gwahior	...	...	1.52	"
5.	Lucknow	...	...	0.88	"
6.	" (duplicate)	...	...	0.79	"
7.	Simla	...	...	0.89	"
8.	Karmuṣa	...	...	0.60	"

NARCOTINE.—Narcotine, having practically no medicinal value, has hitherto not been made the subject of much chemical investigation. Now, however, that cotarnine, one of its oxidation products, has been finding a ready market, the parent alkaloid is also receiving attention. Statements about its solubility, recorded in several places, are neither correct nor complete. Its solubility has therefore been studied in the same way as that of codeine in certain cases, and is considered to be of some practical importance.

Few methods of estimating narcotine have been described, and these are not only incomplete but decidedly erroneous. Henry ("Plant Alkaloids," p. 205) states that "narcotine is usually estimated by extracting the dried opium with dry ether or benzene, and shaking the solution with ammonia, which removes narceine. The narcotine left on distilling off the ether or benzene is dried and weighed." "Allen's

Commercial Organic Analysis" (Fourth Ed., Vol. VI., p. 401) states that "narcotine may be extracted from dried opium by ether or benzene." The narcotine thus obtained will not only contain small quantities of other alkaloids, but also the bulk of the opium wax (*cf.* Rakshit, *ANALYST*, 1918, 43, 321); Plugge's method (*ANALYST*, 1887, 12, 197) for the quantitative separation of narcotine from its hydrochloric acid solution by the addition of a concentrated solution of sodium acetate can hardly be considered as correct, as the alkaloid is appreciably soluble in the dilute acetic acid (see table above) liberated in the solution.

SOLUBILITY OF NARCOTINE IN GRMS. PER 100 C.C. OF SOLUTION AT DIFFERENT TEMPERATURES.

Solvents.				20° C.	30° C.	40° C.	50° C.	60° C.	70° C.	80° C.	90° C.	100° C.
Acetone	...	...	...	4.9	6.3	7.5	9.4	—	—	—	—	—
Benzene	...	...	...	3.4	5.0	6.0	7.5	10.6	14.3	19.3	—	—
Toluene	...	...	...	2.2	2.5	4.0	4.8	7.2	9.4	12.1	17.9	20.8
Ether	...	...	...	0.4	0.6	—	—	—	—	—	—	—
Chloroform	...	...	...	45.9	48.1	50.9	53.4	—	—	—	—	—
Petroleum spirit (b.-pt. 82° C.)	...	...	...	0.02	0.02	0.06	0.07	0.16	0.18	—	—	—
Petroleum spirit (b.-pt. 93° C. to 127° C.)	...	...	...	0.01	0.05	0.09	0.13	0.15	0.21	0.29	0.51	0.64
Alcohol	100	per cent.	...	0.40	0.60	0.90	1.20	2.20	3.20	5.10	—	—
"	95	"	V/V	0.45	0.65	0.97	1.70	2.10	3.30	4.60	—	—
"	90	"	"	0.43	0.60	0.90	1.7	2.1	3.3	4.3	—	—
"	80	"	"	0.32	0.48	0.67	1.29	1.6	2.3	4.2	—	—
"	70	"	"	—	0.32	0.55	0.79	1.3	2.0	4.1	—	—
"	60	"	"	—	0.22	0.29	0.49	0.74	1.35	1.8	—	—
"	50	"	"	—	0.18	0.25	0.32	0.49	0.78	1.06	—	—
"	20	"	"	—	0.07	0.10	0.08	0.09	0.10	0.17	—	—
Ammonia	10	"	W/V	—	—	0.01	0.03	0.04	0.06	0.10	—	—
"	5	"	"	—	0.05	0.02	0.04	0.04	0.04	0.04	0.05	0.05
"	1	"	"	—	—	0.01	0.01	—	—	—	—	—
Acetic acid	20	"	"	—	—	7.35	8.10	9.80	10.7	11.0	—	—
"	10	"	"	—	—	3.10	3.5	3.9	4.0	4.0	—	—
"	5	"	"	—	—	1.50	1.50	1.60	1.70	1.80	—	—
"	1	"	"	—	—	0.40	0.44	0.45	0.52	0.59	—	—
Water	...	...	...	—	0.03	0.07	0.10	0.10	0.10	0.10	0.15	0.20

A method has been worked out and has given satisfactory results during the last four years in this laboratory: Ten grms. of dry opium powder and 5 grms. of fresh slaked lime are triturated in a mortar for half an hour and then transferred to a conical flask, the mortar being rinsed with about 10 c.c. of benzene, and the mixture is shaken well with 100 c.c. of benzene during half an hour. The clear benzene is decanted into a separator, and the residue in the flask twice more extracted with benzene, 50 c.c. being used each time. The total benzene extract is vigorously shaken with 100 c.c. of strong ammonia solution for half an hour, which results in the formation of a permanent white emulsion. This is transferred to a basin and

heated on the steam bath until the benzene has completely evaporated, leaving a soft yellowish residue on the surface of the aqueous ammonia layer. This residue is filtered off and washed until the filtrate ceases to give any odour of ammonia, and is then treated, together with the filter-paper, with 100 c.c. of 1 per cent. hydrochloric acid on the steam bath, when a pinkish solution is obtained. This acid solution is filtered into a separator, the residue washed with a small quantity of water, 100 c.c. of benzene introduced, the solution then made alkaline with ammonia and again very faintly acid with dilute acetic acid, and well shaken for ten minutes. After separation of the benzene the extraction is once more repeated, this time with 50 c.c. of benzene. The benzene extract is filtered and evaporated, and the residue dried and weighed as narcotine. The alkaloid thus obtained has been proved to be practically pure narcotine. The following are typical results of analyses by this process :

Sample.				Narcotine.	
Benares opium	...	...	...	...	6.4 per cent.
Ghazipur "	...	...	...	...	3.6 "
Karmansa opium (received in 1920)	...	...	...	...	1.4 "
Gwalior "	...	...	...	...	2.8 "

The author desires to express his best thanks to Mr. F. D'Costa for analysing many samples by the above process.

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NOTES.

The Editor desires to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

FORMULÆ FOR DILUTING ACIDS, ETC., TO A DESIRED STRENGTH.

For calculating the amount of water to be added to, say, 1 litre of nitric acid of sp. gr. 1.42 to reduce it to *e.g.*, sp. gr. 1.20, the only formula required is $W = VS$, where W represents the weight of a body or liquid with volume V and sp. gr. S . The necessary data are then obtained from sp. gr. tables.

If x represents the volume in c.c. of the diluted acid,

$$1000 \times 1.42 \times \frac{69.80}{100} = x \times 1.2 \times \frac{32.36}{100},$$

and
$$x = \frac{1420 \times 0.698}{1.2 \times 0.3236} = 2552.4 \text{ c.c.}$$

Hence, the required amount of water to be added is : $2552.4 - 1000 =$ (say) 1552 c.c. If contraction takes place during admixture the strong solution must be diluted with water, with cooling if necessary, until the required volume of liquid at 15° C. is obtained. Obviously, the above result also gives the amount of water required to dilute the strong acid to a strength of 32.36 per cent. This simple procedure, which depends on first principles only, is preferable to a formula which is not always remembered.

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THE IDENTIFICATION OF PECULIAR CONSTITUENTS OF FÆCES.

Substances alleged to have been found in fæces, when submitted for identification, call for some caution, as there is a type of patient that thinks it humorous to deceive his medical attendant and another type that malingers. In the following three cases, however, all the evidence pointed to the foreign bodies being genuine constituents of the excretions.

I. A specimen was sent with a request for information whether the creature was an intestinal parasite. It had an arch-shaped body with a tiny anterior segment and ten other segments. Along the dorsal or outer curvature the length was 1 inch, whilst the lower curvature measured $\frac{3}{4}$ -inch. The posterior segment was continued as a tail $\frac{1}{10}$ -inch long, bent at a pronounced obtuse angle at $\frac{1}{3}$ -inch from the body. It was identified as "the rat-tailed larva" of *Eristalis*, a fly of the family *Syrphidae*, which comprises the Hover Flies or Common Drone Flies. The larvæ are said to live in liquid manure and foul water, and have been seen on watercress. Fantham, Stevens, and Theobald ("Animal Parasites of Man," 1916) only mention one specific case of human infection—that recorded by Austen—where in the autumn of 1907 a number of larvæ of *Eristalis tenax* was passed *per rectum* by a woman in Hampshire, who had recently arrived from France. The patient had eaten a considerable quantity of watercress before leaving France.

The larva was described about 1750 by Réaumur, and was called by him *le vers à queue de rat*. Henry Baker ("Of Microscopes," 1785) obtained a larva from a cistern of rain-water in August, and he observes the flies to be more common in France than in England. This author mentions the power the larva has of lengthening and retracting its tail, which he found could reach a length of four inches on occasion. This is of importance, because, until the telescopic nature of this organ is known, there is difficulty in identifying with illustrations a dead larva possessing a partly retracted tail.

The present specimen came from Kent at the beginning of October, 1921.

II. Some pale cream-coloured shreds were submitted to ascertain whether tapeworm was present. Microscopical examination indicated nothing in the contour of the fragments to suggest tapeworm, and no ova could be found. There was, instead, a matrix of some translucent substance with pieces of talc-like material embedded therein. When ignited the specimen did not emit a nitrogenous odour, but left an appreciable amount of ash containing 61 per cent. of silica, some magnesium, and traces of iron and aluminium. It also contained calcium and phosphate distinct from the magnesium silicate. Enquiry showed that the patient was in the habit of taking twenty saccharin tablets a day, which presumably formed the source of the talc, though a *post hoc* explanation of the presence of an article in fæces is unconvincing, because from time to time instances occur of masses remaining in the intestines for years before digestion.

III. This specimen consisted of five stones, each of which had a brown waxy centre and a creamy-brown powdery coating. The total weight was 1.4 grms. They had the following percentage composition: Moisture, 6.5; mineral matter, 4.0; mucin, 1.7; vegetable tissue, 2.8; resinous matter, 80.3; and other substances, 4.7. The bran of grain and a trace of cork tissue were found among the vegetable debris. No notable amounts of cholesterol, bile-pigments, or calcium carbonate were present. The extracted resin was insoluble in water, but soluble in ether, in alcohol, and in quite a small quantity of acetic acid. These results would appear to exclude euonymin and podophyllum resin, which had been prescribed, as sources of origin. Later information suggested that it might be a residue from one or other of the drugs prescribed for urinary antiseptis. A trace of iron and a faint trace of zinc, but no copper, were present.

W. PARTRIDGE.

ANALYSIS OF A SOUTH AMERICAN COPPER AXE HEAD.

Very few analyses of South American copper axe heads collected in the burial-grounds of the Indians of Peru and of Ecuador have been published. One by Humboldt indicates that the hardening of the copper making it suitable for the purpose in view was accomplished by the addition of 0.4 per cent. of tin, an addition probably of accidental origin rather than of design. A South American hatchet collected by one of us (E. K. R.), and stated to have belonged to an Inca Indian (Inca Pirqua), was submitted to analysis with the following results:

The metal possessed a coppery lustre, and was tough and malleable rather than brittle. The form of the hatchet suggested that it had been hammered into a plate and the haft burred up at the edges. It contained copper, zinc, iron, and small traces of silica, whilst tin, lead, and nickel were absent.

For the quantitative analysis the metal was dissolved in nitric acid and the copper estimated electrolytically. Iron was estimated colorimetrically in an aliquot portion of the residue and the zinc precipitated as sulphide, ignited and weighed as oxide, the method and precautions adopted by Sir W. Crookes being followed and allowance being made for the small quantities of iron. The silica was present in quantities too small for accurate estimation. Duplicate analysis yielded the following results: Copper, 96.42 and 96.45; zinc, 3.46 and 3.41; and iron, 0.09 and 0.09. Total, 99.97 and 99.95 per cent.

These results are particularly interesting in the fact that they indicate the material of the axe head to be a brass and not copper. Berthelot (*Comptes rend.*, 1893, 116, 161; 1894, 118, 764; 1897, 124, 328) found that the earlier copper articles of Chaldea and Egypt (*circa* 4000 B.C.) were made of pure copper, whilst the earlier copper-tin articles have been attributed by Coffey (*J. Anthropol. Inst.*, 1901, 31, 265) to the accidental use of a tin containing copper ore, and not to foresight on the part of the maker. Brass articles have always been considered to belong to a very late period. It may be noted that there was no reason to suspect the genuineness of the axe head in question.

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THE NATIONAL PHYSICAL LABORATORY.

REPORT FOR THE YEAR 1920.*

THE Report on the work of the National Physical Laboratory for 1920 numbers 182 pages. It comprises a Report of the Executive Council, giving a general outline of the work of all the departments, a statistical comparison of the tests made in the years ending 1914, 1919, and 1920, a statement of the work proposed for the year 1921-22 and approved by the General Board, a list of the Laboratory staff, a list of the scientific papers published by the Laboratory or communicated to scientific or technical journals, and the detailed Report of the Director for the year 1920. There is also a Report of the Advisory Committee of the William Froude National Tank, in which fifty-three ship models have been tested during the year.

PHYSICS DEPARTMENT: HEAT DIVISION.—Systematic work on the thermal conductivity and specific heat of cold storage heat-insulating materials has been carried out, and a series of reports presented to the Engineering Committee of the Food Investigation Board. Data for the thermal conductivity of all the materials commonly used for cold storage installation have been determined. The influence of baffles,

* Copies of the Report may be obtained from H.M. Stationery Office, Kingsway, London, W.C. 2. Price 5s. net.

etc., has been studied in detail, as well as the velocity and temperature distribution of the convection stream in the vicinity of a heated surface. For these measurements a convenient form of hot wire anemometer has been devised, which combines the functions of a resistance thermometer and an anemometer. Another research involved the determination of the specific heat of poor conductors, such as earth, slag, wool, cork, and charcoal. Experiments on heat loss from surfaces were undertaken for the British Portland Cement Association, and the coefficients of expansion of metals at high temperatures have been determined by means of two forms of apparatus specially devised for the purpose. During the year twenty-one Abel-Pensky flash-point apparatus have been submitted for testing.

THERMOMETRY DIVISION.—In the course of the year ending September 20, 1920, over a million and a half thermometers have been tested. The number of rejections has fallen to less than 2 per cent., the highest percentage with certain firms being 5 to 8, whilst it was over 30 per cent. in the case of a number of instruments of foreign manufacture.

RADIOLOGY DIVISION.—The primary British standard of about 30 mgrms. of hydrated radium bromide has now been supplemented by two secondary standards containing about 10 and 13.5 mgrms., which will be used ordinarily for the standardisation of quantities sent for test. Experiments on radium luminous compounds have been continued, and a research on the absorption and scattering of γ -radiation in metals is in progress.

OPTICS DIVISION.—Comparisons of different methods of refractometry have been made, and a method has been devised whereby check measurements can be made without the use of a spectrometer.

ELECTRICITY DEPARTMENT.—Much work has been done in connection with radio standards, the measurement of effective resistance at radio frequencies, etc., and various researches on valves have been continued. In addition to the testing of magnets and magnetic materials, a method has been devised for measuring the effective permeability and energy losses in thin sheet iron at radio frequencies.

PHOTOMETRY DIVISION.—Experiments have been made on ships' navigation lamps, miners' lamps, and motor-car head lights for Government Departments, and other experiments are in progress with a view to the specification of suitable lighting schemes for public buildings and offices.

METROLOGY DEPARTMENT.—Exclusive of tests of gauges for the Ministry of Munitions and of taximeters, 20,915 routine tests have been made, an increase of nearly 4,000 over the previous year. Coefficients of expansion of concrete blocks, as used in building houses, of "rustless" steel, glass, ebonite, etc., have been determined.

Physical Instruments.—In testing balances the instrument is placed $5\frac{1}{2}$ m. from the source of light and the scale on which the image is read, and the effect of radiation of heat into the balance cases has been reduced to a minimum. A set of solid rock-crystal weights (200 grms. to 1 grm.) has been obtained, and will be used as reference standards. Progress has been made in the re-verification of the Laboratory standards of mass by comparison with the Sèvres standards.

(a) *Glass Volumetric Apparatus.*—The number of vessels tested was 2,556, as compared with 1,455 in the previous year and 388 in 1917-18. During the year specifications for volumetric glassware used in milk analysis have been drawn up and approved by a conference comprising manufacturers, users, and a number of expert dairy chemists. The South African Government has agreed to recognise the National Physical Laboratory as one of the official testing stations for the volumetric glassware to be used in dairy chemistry in dairies, butter factories, etc. Most of the vessels are tested gravimetrically, and vessels which pass the test are etched with the Laboratory monogram by means of special apparatus. An investigation on the difference in volume of milk and of water delivered by pipettes under various conditions has proved valuable in drawing up specifications for milk pipettes, and has

indicated the necessity of modifying the usual type of instrument used. An investigation on the effect of varying the delivery and drainage time of pipettes of various capacities has also been completed. A volumetric method of testing butyrometers by the use of mercury has been devised.

(b) *Hydrometers*.—The number of hydrometers tested during the year was 1,481, an increase of 72 per cent. on the previous year. A detailed investigation is in progress relating to the effect of surface tension on the indication of hydrometers. New standard specific gravity hydrometers have been ordered, extending the range of the Laboratory standards from 1.85 to 2.00.

ENGINEERING DEPARTMENT.—A series of researches on lubricants and lubrication has been carried out for the Committee on Lubricants of the Department of Industrial and Scientific Research (*cf.* ANALYST, 1921, 453), and other investigations include one on the effect of pressure and temperature on the production of detonation in internal combustion engines. Various building materials, including concrete blocks, have been tested for strength, but those tested have indicated no great advance upon the usual building bricks.

DEPARTMENT OF METALLURGY.—Much work has been done in connection with light alloys, and the investigation of the constitution and physical properties of alloys of zinc containing moderate amount of copper and aluminium has been completed. Considerable progress has been made with the preparation of analytically standardised steel samples, in conjunction with a Research Committee of the Iron and Steel Institute. Samples of the millings have been sent to the co-operating analysts, and it is hoped that the standardised samples will be available for public issue in the course of a few months. The general chemistry of the Department has included analytical work on special bricks in connection with the housing schemes of the Ministry of Health.

Optical Glass Research.—The study of pot attack and the methods of producing suitable pots has been continued. Experiments on the viscosity of glass at various temperatures are in progress, and an apparatus for making such measurements at high temperatures has been devised.

Aeronautical Chemistry.—The differences between various acetates cannot at present be related to their chemical constitution, and experiments are in progress to ascertain to what extent variations in the viscosity during saponification would indicate the extent of homogeneity of the product. The study of hydrometric methods required in connection with the effects of humidity on fabrics and dopes has been continued. The Shakespear permeameter, calibrated in the Department, is now used for routine tests of the permeability of balloon fabrics by hydrogen. Experiments as to the suitability of glycerol as a standard in the viscosimetry of viscous solutions, such as dopes, have shown that commercially pure samples of glycerol exhibit considerable variation in viscosity, even when their specific gravities have been adjusted to be identical. Certain chemical tests have therefore been devised which will enable approved samples to be used by different workers so as to obtain concordant results in the examination of cellulose acetate, etc.



REPORT OF THE GOVERNMENT CHEMIST UPON THE WORK OF THE GOVERNMENT LABORATORY.*

FOR THE YEAR ENDED MARCH 31, 1921.

THE total number of samples examined during the year was 308,675, as compared with 368,898 in the preceding year, the decrease being accounted for by the diminution in the number of samples tested locally at chemical stations on the coast. The samples examined at the central laboratories in London amounted to 204,653, as against 199,388 in the previous year.

CUSTOMS AND EXCISE: Beer.—The total number of samples examined in connection with the duty on beer was 28,167, being an increase of nearly 3,000 samples. For the purpose of checking dilution and adulteration, 854 samples of beer were examined, as compared with 21 in the previous year. In 34 cases there was evidence of dilution, which in 6 cases was equivalent to the addition of over four gallons of water per barrel.

Non-Alcoholic Beers, Herb Beers, and Beer Substitutes.—Of the 61 samples of non-alcoholic beers examined, 17 contained between 2 and 5 per cent. and 1 over 5 per cent. of proof spirit. The number of samples of herb beers, ginger-beers, etc., examined was 69, of which 13 contained between 2 and 5 per cent. of proof spirit.

Imported Beer.—The importation of beer, mostly lager beer, is still insignificant, the number of samples examined being only 426, as against nearly 10,000 in the year ended March 31, 1914.

Examination of Beer and Brewing Materials for Arsenic.—The number of samples of beer, worts, and materials of all kinds tested was 1,258, of which 40 were found to contain arsenic in excess of the limits laid down by the Arsenic Commission. Of the 692 samples of malt and sugar examined only 1 contained arsenic in excess of the limit, whilst the limit was exceeded in the case of two samples out of 300 of beer and wort.

Cider and Perry.—Six samples of cider out of 79 examined were found not to consist solely of fermented apple-juice, and were accordingly charged as spirituous preparations.

Table-Water Duty.—Of the 58 mineral waters, 35 proved to be medicinal, and 23 were liable to duty. Of the 654 cordials, syrups, etc., only 19 contained more than 2 per cent. of proof spirit. Three of the 42 samples of "non-alcoholic" wines contained more than 2 per cent. of proof spirit, the highest being 3·8.

Spirits.—The declared gravity of the fermented wash was found to be understated in 41 cases out of the 395 examined.

Fusel Oil.—Fusel oil, whether imported or home produced, generally contains ethyl alcohol as an unavoidable impurity: but duty is not charged unless upwards of 15 per cent. of proof spirit is present, which was the case in 19 of the 88 samples tested.

Grogging.—In 5 cases evidence of the spirit having been extracted from the wood of the cask was obtained.

Illicitly Distilled Spirits.—Twelve suspected samples were examined, and in several instances the analytical results furnished evidence of illicit distillation. In 2 cases prosecutions were instituted and convictions obtained.

British Spirituous Preparations Exported.—During the year 22,184 samples of medicinal spirits, tinctures, perfumes, etc., were examined, to check the exporters' claims for drawback on the spirit used in their manufacture; in addition, 3,456 liniments, hair washes, etc., were tested for methylated spirit, the use of which precludes the right to drawback on exportation. Methyl alcohol was detected in one sample, and in 392 cases the alcoholic strength had been overstated.

Imported Spirits and Spirituous Preparations.—The number of samples of imported spirits was 2,742, an increase of about 400 samples on the previous year. Of 59 samples of methylic alcohol, 22 were found liable to duty on the ground of purity.

Duty-free Spirits, including Methylated and other Manufacturing Spirits.—The naphtha, represented by 720 samples, was approved as fit for methylating purposes, whilst 13 samples were reported as unsuitable.

Wines.—In 8 cases only was the alcoholic strength in excess of 42 degrees, probably due to overfortifying. Sherries and Marsala were usually well over 30 degrees. The total number of samples examined was 71,159, as compared with 143,544 in the preceding year, when exceptionally heavy imports of wine followed the Armistice.

Sugar, Glucose, and Saccharin.—The total samples examined number 24,334, or 32,332 including the outport chemical stations. Tests for saccharin were applied to 274 samples of imported substances.

Tobacco.—The percentage of moisture was determined in 542 samples of imported unmanufactured tobacco, whilst 10 samples were submitted for decision as to whether the tobacco was liable to duty as "leaf" or "strip." Twenty-seven samples of manufactured tobacco were submitted for classification, 12 of which contained ingredients rendering them inadmissible for importation. More than half of the 147 samples of home-grown leaf tobacco came from Methwold, Norfolk, where the experimental culture is being assisted by the Ministry of Agriculture. The percentage of moisture was determined in 6,321 samples and the oil in 457 samples of manufactured tobacco for home consumption. The samples of tobacco exported on drawback numbered 11,514, as compared with 13,797 in the previous year. Advantageous treatment in the matter of sampling is accorded to those manufacturers who furnish declarations as a basis for the payment of drawback. During the year 19,577 samples of tobacco stalks for drawback purposes, and 5,648 samples of offal snuff, shorts, and smalls, for denaturing, abandonment, the manufacture of nicotine or sheep dips, or on exportation, were examined. Seven samples of various herbal smoking mixtures were found to be free from any admixture of tobacco.

Tea.—The total number of samples examined was 32,176, which is far in excess of those received in any previous year. This is due to the importation of numerous small shipments in the form of dust or otherwise of doubtful quality. Some samples contained foreign seeds derived from grasses, which probably grew in or near certain tea-gardens. The samples reported against numbered 10,025, or 31 per cent. of those examined. The presence of the foreign substances in these was attributed to accidental admixture with small quantities of sand from the soil or to warehouse impurities. The number of samples of tea denatured with lime or asafoetida for use in the manufacture of caffeine was 749.

Coffee and Coffee Substitutes.—Of the 1,610 samples examined only one was incorrectly declared.

Cocoa and Chocolate.—The amount of raw cocoa was estimated in 6,613 samples either on importation or (for drawback) on exportation. Fixed rates, revised periodically after analysis, have been established for goods regularly imported or exported, and most of the firms engaged in the trade have taken advantage of these facilities. A method of estimating a substitute for cacao butter in chocolate has been devised in the laboratory (ANALYST, 1921, 229). Eight hundred and eighty-four samples of chocolate confectionery were found to contain spirit.

Imported Synthetic Dyestuffs.—Ninety samples of imported colours, inks, perfumes, etc., were examined to ascertain whether they contained synthetic dyes or intermediate products, the importation of which is prohibited except under licence.

Matches.—In none of the 67 samples submitted was any evidence of the presence of white phosphorus obtained.

Medicine Stamp Duty.—Seventy-eight samples were examined to establish their identity or in connection with import restrictions and the opium and cocaine regulations.

Hydrometers, Saccharometers, etc.—During the year 2,247 hydrometers, saccharometers, and graduated vessels for use at distilleries, breweries, glucose factories, and in the Surveying Department have been calibrated before being issued.

ADMIRALTY.—Samples of food on tender have been submitted for report as to conformity with specification. Other samples included ferrous and non-ferrous metals of all kinds, fuel oils, rubber washers, soaps, fabrics for sail cloths, and paints. The total number of samples was 739.

AIR MINISTRY.—Various metals and alloys have been examined, and investigations as to corrosion due to greases have been made. The number of samples dealt with was 185.

MINISTRY OF AGRICULTURE AND FISHERIES: *Imported Dairy Produce and Margarine.*—Nine samples of imported butter were satisfactory. Of 1,100 samples of margarine 19 were found to contain more than 16 per cent. of water. There was no evidence of excess of butter in any of them. All the samples of cheese were satisfactory. The fat varied from 9·3 to 39 per cent. Some of the cheese had therefore been prepared from milk from which a portion of the fat had been removed, but as there are no regulations relating to the marking of skimmed or partially skimmed cheese, no exception could be taken to the importation. Some of the samples of imported cream contained just under 9 per cent. of fat, but there was no evidence of dilution with water. Four hundred and thirty-one samples of condensed milk, including milk powders, were examined. In nine cases the condensed milk had been made from skimmed milk, but this was not indicated on the package. One of the samples was labelled "Centrifuged Separated Milk"—a description which was not considered a sufficiently clear description.

Butter and Margarine Samples taken at Factories in this Country.—The inspection in Great Britain has not been resumed since the war. Of the 155 samples from Ireland 20 contained more than 16 per cent. of water.

Sheep Dips.—Of 86 samples prepared in accordance with formulæ approved by the Ministry, 36 were rejected as being defective.

Water and Pollution of Rivers.—Twenty-four samples of river water and effluents were investigated. Two kinds of pollution appear to be extending: effluents from milk and cheese factories, and from suction gas plants using wood and sawdust fuel. The latter effluents contain products of the distillation of wood, and are toxic to fish, apart from their deoxygenating effect on well-aerated water. It has been ascertained that considerable purification of such effluents can be readily effected. Trustworthy information as to the proportions of toxic constituents in tar extracts and washings has now been obtained, and details of the methods of examination are in course of publication. In view of a recent legal decision the composition of road drainage and tars is a matter of extreme importance.

Sea Water.—An investigation as to the influence of the salinity of sea water upon fish is in progress. In order to make the results comparable with those obtained by the different countries concerned in the work, a prepared standard sea water is issued from a central bureau, and a uniform process of analysis employed. The salinity of 4,653 samples, taken at recorded depths, temperatures, geographical positions, etc., has been determined during the year.

Fertilisers and Feeding Stuffs Act.—Reports have been given on 20 fertilisers and 10 feeding stuffs in cases where there has been a discrepancy between the statement of the seller and the finding of the official analyst. In most cases the fertilisers contained less nitrogen, phosphates, or potash than the guarantee. In other cases samples sold as fish guano contained very little fish and were without appreciable fertilising value.

The feeding stuffs consisted of milling offals and various kinds of meals. In some cases there was a deficiency of oil or albuminoids compared with the guarantee on the label; in other cases foreign substances (*e.g.*, corn cockle, a poisonous weed seed, and rice husk). Some meals for feeding pigs contained nearly 8 per cent. of sand, whilst a "fish meal" consisted mainly of cereals with less than 10 per cent. of fish or meat meal. In all cases there was substantial agreement with the results of the agricultural analysts who had condemned the samples.

Miscellaneous Articles.—Two samples of exhausted raisins intended for manure contained: Water, 70.57 and 72.94; organic matter, 23.51 and 17.48; mineral matter, 5.92 and 9.58; nitrogen, 0.48 and 0.59; phosphate, 0.38 and 0.47; and potash, 0.96 and 1.40 per cent. The material was thus similar in general fertilising value to farmyard manure.

China Clay in Feeding Stuffs.—Another sample contained china clay, the addition of which to fine meals might escape detection until a chemical analysis was made. The following methods of detecting very small quantities were devised and communicated to the Ministry for circulation: (1) The fine dust passing through a sieve 100 meshes to the inch is ignited and examined microscopically with polarised light and crossed "Nicols." (2) The suspected sample is shaken with ether, and after the tube has been stood for a few minutes, the ethereal layer, which will contain the kaolin and starch in suspension, is decanted and evaporated, the residue ignited, and the ash examined microscopically. If kaolin be present the ash will consist mainly of fine crystals which show their felspathic origin.

The Composition of Oysters taken from selected ground at regular intervals during a year was determined for the Fisheries Department. In the fresh condition oyster meat contains: Water, 76 to 82; fat, 1.5 to 2.5; proteins, 9 to 12; carbohydrates, 4.5 to 8; and mineral matter, about 2 per cent. The carbohydrate matter consists mainly, if not wholly, of glycogen. The amount of glycogen estimated immediately on opening the oyster corresponds approximately with the total carbohydrates, whereas in the dried oyster meat it is only 50 to 60 per cent. of the total carbohydrates present. The dry ash-free solids contain the following proportion of food constituents: Fat, 8.5 to 11.9; proteins, 50.2 to 64.3; and carbohydrates, 22.9 to 39.4 per cent.

GEOLOGICAL SURVEY.—Fifteen samples of water from boreholes were examined. Some contained both barium chloride and sulphates, the co-existence of which in dilute brine raises interesting physico-chemical questions.

HOME OFFICE.—Ten samples have been tested for opium and certain drugs under the Dangerous Drugs Act, whilst 89 samples were examined for the police authorities. These included food mixed with phosphorus, paper saturated with petrol from premises which had been on fire, and substances found on persons alleged to be dealing illicitly in cocaine.

The examination of lead glazes, enamels, etc., affecting the health of workers in factories has been continued, and experiments were made for the Factory Department to ascertain the amount of lead and other metals discharged into the air during soldering and lead burning.

POST OFFICE.—The paper, pigments, and gum used in the manufacture of stamps were systematically examined for quality and freedom from deleterious substances. As in previous years, investigations have been made in cases of suspected fraudulent use of postage and fiscal stamps. A special inquiry was made as to the disintegrating effect of certain inks on rubber stamps, and, as the result, certain constituents of such inks were eliminated.

BOARD OF TRADE: Lime and Lemon Juice for the Mercantile Marine.—Of 228 samples of crude lime juice submitted only one was found unsatisfactory. The quantity of fortified juice approved was 25,926 gallons.

Potash Branch.—Two hundred and thirteen samples of potash from supplies

from Germany were analysed. Each original sample had been divided into three parts, one of which was analysed in Germany, whilst the third was retained in a neutral country for analysis in case of dispute. There were only a few cases in which the Germans asked for the analysis of the third portion, and in each instance there was substantial agreement with the analysis here.

WAR DEPARTMENT.—The number of food samples examined was 3,249. Returned stores were also examined as to their fitness for use. The amount of tin in canned food which had been in store for a year or two was estimated in suspicious cases, and, if excessive, the foods were destroyed.

OFFICE OF WORKS, LONDON.—In addition to the examination of materials of all kinds for the public service, special investigations were made as to the suitability of paints and varnishes for protecting surfaces of metal and wood for external work. The effect of town atmosphere on building stones was demonstrated, the destructive agents being mainly sulphuric or sulphurous acids. It was also shown that ordinary domestic soot could disintegrate limestone, owing to the acid, ammonium sulphate, etc., in it. It has been established that the addition of small amounts of salt or calcium chloride to prevent freezing of the water for making cement in cold weather has no deleterious influence on the properties of the cement.

SALE OF FOOD AND DRUGS ACTS.—Thirty-six samples referred by magistrates were examined during the year, including 33 milks, 1 sausage, 1 butter, and 1 lemon squash. In 31 cases the results were in agreement with those of the analyst for the prosecution. Four of the other samples were milks in an advanced stage of butyric fermentation; with regard to these no certificate was given. The only case in which there was direct disagreement with the analyst was in connection with a sample of butter; this was alleged to contain excess water, but was found not to contain more than the legal amount. The sample of "lemon squash" contained only a small proportion of lemon constituents, the acidity being mainly due to the presence of free phosphoric acid.

Five samples were referred in cases arising from the Beer and Spirits (Prices and Description) Orders of the Food Controller.



UNITED STATES DEPARTMENT OF AGRICULTURE.

FOOD INSPECTION DECISION 182.

CITRUS FRUITS.—The following definitions and standards for grape fruit and oranges were adopted by the Joint Committee on Definitions and Standards, March 23, 1921, and were approved by the Association of American Dairy, Food, and Drug Officials, October 7, 1920, and by the Association of Official Agricultural Chemists, August 19, 1921:

Grape fruit, pomelo, is the sound, mature fruit of *Citrus grandis* Osbeck. The juice of the mature fruit contains not less than seven parts of soluble solids to each part of acid calculated as citric acid without water of crystallisation.

Orange (common, sweet, or round) is the sound, mature fruit of *Citrus sinensis* Osbeck. The juice of the mature fruit contains not less than eight parts of soluble solids to each part of acid calculated as citric acid without water of crystallisation.

The foregoing definitions and standards are adopted as a guide for the officials of this department in enforcing the Food and Drugs Act.



ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Estimation of Chlorine in Milk without Calcination. J. Werder. (*Mitt. Lebensm. Hyg.*, 1921, 12, 37-38; *Chem. Abstr.*, 1921, 12, 1950.)—Fifty c.c. of the milk are boiled for thirty minutes with 25 c.c. of nitric acid (sp. gr. 1.15). After cooling, 20 c.c. of $\frac{N}{10}$ silver nitrate solution are added, and the mixture heated on a water-bath for fifteen minutes and filtered. Any fat remaining in the filtrate is extracted with 20 c.c. of ether, after which 2 c.c. of saturated ferric ammonium alum solution are added, and the excess of silver titrated with $\frac{N}{10}$ ammonium thiocyanate solution. The results quoted agree well with those obtained by the usual method.

T. J. W.

Calcium Content of White of Egg. H. Kreis and J. Studinger. (*Schweiz. Apoth. Zeit.*, 1921, 59, 193-196; *Chem. Abstr.*, 1921, 15, 2315.)—The statement of Rözsenyi's (*Chem. Zeit.*, 1904, 620), that the amount of calcium oxide in the ash of the whites of eggs which had been kept twenty-four hours in lime water increased from 1.83 to 2.08 per cent., is inconclusive, since the same amount may be present in untreated eggs. Should the yolk membrane break and the yolk (11.54 to 12.32 per cent. CaO in the ash) mix with the albumin higher results for the latter will naturally be obtained. The whites of eggs which had been kept for nineteen days in lime water yielded 0.8 per cent. of ash containing 1.18 per cent of calcium oxide. In another test the fresh eggs showed 0.75 per cent. of ash, with 2.64 per cent. of calcium oxide; after one month, 0.8 per cent. (2.04 per cent. CaO); three months, 0.76 per cent. (1.44 per cent. CaO); and six months, 0.73 per cent. (CaO, 1.12 per cent.). The albumin of fresh eggs yielded ash containing 0.59 to 4.25 per cent. of calcium oxide; there was no absorption of calcium after they had been kept for months in lime water, and the specific gravity of the eggs was unaltered, but the yolk membrane was rapidly weakened. These results show that the amount of calcium in the ash of the albumin affords no proof of the preservation of the eggs in lime water.

Caviare and Caviare Substitutes. P. Buttenberg. (*Chem. Zeit.*, 1921, 45, 1025-1026.)—During and since the war a number of preparations of the roe of salmon, cod, sole, herring, and other sea fish, has been put upon the market as "caviare," in place of the real (Russian) article, composed of the eggs of the sturgeon preserved by freezing, salt, or boric acid. (In Russia preservation other than by freezing or salting is forbidden.) The eggs of all the above fishes are distinguishable from those of the sturgeon by their smaller size; moreover, those of the sturgeon are of a characteristic dark grey or silver grey colour. It is urged that such substitutes should be compulsorily labelled with the name of the fish from which they have been prepared, and not described simply as "caviare"; also, that

artificial colouring to imitate the true caviare colour should be forbidden. Artificial colouring can easily be detected by shaking the caviare with water and alcohol; artificial dyes colour the liquid. The condition of the sample is best determined by estimating the acidity and the ammonia liberated on distillation with magnesia. The dark-coloured fish roes often show an acidity of 17 to 19 mgrms. and an ammonia content of 190 mgrms. per kilo, but there are other preparations from which the ammonia has been almost completely removed by washing with water.

H. E. C.

Impurities of Cacao. E. Wilczek. (*Schweiz. Apoth. Zeit.*, 1921, **59**, 120-126; *Chem. Abstr.*, 1921, **15**, 2315.)—An indication of the origin of cacao and of its value may be obtained from the nature of the impurities normally present. The impurities characteristic of samples from tropical America include: *Cocos* spec., *Astrocaryum* spec., *Maximiliana* spec., *Euterpe oleacea*, *Phytelephas macrocarpa*, *Bertholletia excelsa*, *Dipterix odorata*, *Vittellaria mammosa*, *Mammea americana* and *Cupuassu*. In the case of Cameroon cacao the following impurities are distinctive of the origin: *Elæis guineensis*, *Cocos nucifera*, *Amomum melaguetta* and *grandiflorum*, *Cola vera* and *acuminata*, *Arachis hypogæa*, *Voandzeia subterranea*, *Physostigma cylindrosperma*, *Pentaclethra macrophylla*, *Monodora myristica* and *angolensis*, *Xylopia* spec., and *Carapa procera*.

Estimation of Reducing Sugars in Lead-Preserved Sugar-Cane Juices. J. B. Harris. (*J. Ind. Eng. Chem.*, 1921, **13**, 925-926.)—To obtain the correct quantity of reducing sugars in samples of sugar-cane juices which have been preserved with lead acetate it is necessary to remove the lead before the sample is filtered. Oxalic acid is the best substance to use for precipitating the lead; the usual neutral salts are unsuitable for the purpose, since their use causes the results for the reducing sugars to be too low.

W. P. S.

Methods for the Analysis of Artificial Honey. A. Behre. (*Chem. Zeit.*, 1921, **45**, 1002-1003.)—The methods for estimating the total dry matter, cane sugar and starch glucose in artificial honey, are reviewed. The refractometer affords the best method for estimating the total dry matter. The process of inversion gives rise to the formation of certain condensation products, which will reduce Fehling's solution and have a specific rotation similar to that of fructose, so that the determination of sucrose and dextrose by the Fehling-Allihn or Clerget processes and by the polarimeter generally gives too high results. For this reason the iodine process is to be preferred for accurate work; but the polarimeter answers satisfactorily for approximate analysis, for which purpose the specific rotation of artificial honey should be taken as -20° . Owing to errors in the sugar estimations, the determination of the so-called sugar-free dry matter is usually quite inaccurate, and, in the present state of knowledge, cannot be made accurate. The sugar-free dry matter sometimes contains lævulinic acid and a little formic acid, and detracts from the food value of the honey. The iodimetric estimation of the sucrose is carried out as follows: Twenty c.c. of a 1 per cent. solution of the honey are diluted to 50 c.c., and 3 c.c. of hydro-

chloric acid (sp. gr. 1.19) are added; the mixture is heated quickly in a water-bath to 69° C., kept at this temperature for five minutes, and then cooled; phenolphthalein is added and 33 per cent. sodium hydroxide solution run in until the colour is pink. This colour is discharged with $\frac{N}{10}$ acid, 25 c.c. of $\frac{N}{10}$ iodine solution are added, then 29 c.c. of $\frac{N}{10}$ sodium hydroxide solution, and the mixture is allowed to stand for 20 minutes, after which time it is acidified with dilute sulphuric acid and titrated with $\frac{N}{10}$ thiosulphate solution. The sucrose is measured by the difference in iodine value before and after inversion, as above (cf. ANALYST, 1921, 368).

H. E. C.

Artificial "Honey." A. Beythien. (*Chem. Zeit.*, 1921, 45, 1026-1027.)—At a conference of chemists connected with the industry the following specifications were adopted: Artificial honey must possess good keeping qualities; it must not yield more than 3 per cent. of ash, must have a honey-like aroma, and may be coloured artificially. The acid used for the inversion must be technically pure, and contain no injurious substance; the free acidity left in the honey must not exceed 2 mgrm. equivalents per 100 grms. The total dry matter must be more than 78 per cent., and the content of uninverted cane sugar must be not more than 25 per cent. The addition of starch syrup and glucose is allowed to a total amount of 20 per cent. if both materials are of a quality specially fitted for human consumption. The product must give a strong reaction in Fiehe's test (ANALYST, 1909, 34, 399; cf. 1908, 33, 397), and the addition of some natural honey as flavouring is allowable. The package must be distinctly labelled "artificial honey."

H. E. C.

Grape-Seed Oil. F. Rabak. (*J. Ind. Eng. Chem.*, 1921, 13, 919-921.)—The variety of grapes grown for the manufacture of grape-juice in the United States is the well-known Concord grape; about 1,100 tons of grape-seeds are obtained annually, as a by-product, in the manufacture of the juice. The seeds yield about 13 per cent. of oil, which may be used for edible purposes. The refined oil has a pale yellow colour, a pleasant odour and taste, and the following physical and chemical characteristics: Sp. gr. at 25° C., 0.9204; solidifying point, -22° to -24° C.; acid value, 0.74; saponification value, 192.2; and iodine value, 135.8. These results differ in many respects from those recorded in literature for other grape-seed oils. The composition of the oil is (approximately): Linolin, 53.6; olein, 35.9; palmitin, 5.2; stearin, 2.2; and unsaponifiable matter, 1.6 per cent.

W. P. S.

Glycerides of Goose-Fat. A. Bömer. (*Chem. Zeit.*, 1921, 45, 1026.)—Goose-fat contains the glycerides of palmitic, stearic, and oleic acids, and not those of margaric acid, as is stated by Klimont and Mayer (cf. ANALYST, 1915, 40, 349). Solid glycerides are present in small quantity as palmitodistearin and dipalmitostearin; liquid glycerides preponderate as triolein and palmitodiolein, together with some steardiolein.

H. E. C.

Detection of Saccharin in Wine. W. Fresenius and L. Grünhut. (*Zeitsch. anal. Chem.*, 1921, 60, 353-359.)—The wine (500 c.c.) is evaporated to a small volume to remove the alcohol, and the residue diluted to 450 c.c. with water, acidified with 5 c.c. of dilute sulphuric acid (sp. gr. 1.11), and then extracted successively with 60 c.c. and two portions of 25 c.c. of ether. After removal of the ether, the wine is evaporated to 200 c.c., cooled, and shaken out three times with 200 c.c. of ether. The bulk of the mixed ether extractions is then distilled, and the last portion thereof evaporated in a porcelain dish. The residue is taken up with 50 c.c. of water and the mixture evaporated to dryness, this process being repeated; the residue is then dissolved in 5 c.c. of $\frac{N}{2}$ sodium hydroxide solution, the solution heated on the water-bath, and 5 per cent. potassium permanganate solution added in small quantities until the pink colour persists for one minute. The liquid is now acidified, and more permanganate added, if necessary, to retain the pink colour for a minute; a dilute solution of sulphur dioxide is next added in quantity just sufficient to decompose the excess of permanganate and dissolve any manganic oxide which may have separated out. The clear liquid is extracted three times with an equal volume of ether; the ether is distilled off, and the last portion evaporated at a low temperature in the presence of 1 c.c. of water. After complete removal of the ether, and cooling, a sweet taste indicates the presence of saccharin, which may be confirmed by evaporating the residue in a silver crucible and fusing it for half an hour at 250° C. with 0.5 gm. of sodium hydroxide. The fused mass is dissolved in water, the solution acidified with dilute sulphuric acid and extracted with ether, and the ether extract evaporated and tested for salicylic acid with ferric chloride. A violet coloration confirms the presence of saccharin.

H. E. C.

Estimation of Camphor in Camphorated Oils. D. A. Wallace and S. B. Plummer. (*Amer. J. Pharm.*, 1921, 93, 600-604.)—The results of experiments described show that the camphor present may be estimated from the loss on heating 5 grms. of camphorated oil at 120° C. for five hours for cotton-seed oil; four hours for olive and arachis oils; and three hours for sesame oil; and allowing for the loss or gain in weight on 4 grms. of the oil without camphor by adding or subtracting for the respective oils, +0.0142, +0.0138, +0.0108, -0.0010. For example, 5 grms. of cotton-seed oil containing 20 per cent. of camphor lost 0.9855 gm. in five hours, whilst 4 grms. of the oil alone gained 0.0142 gm.; the camphor present would therefore be $0.9855 + 0.0142 = 0.9997$ gm. The saponification values of the oils were but little affected by heating at 120° C., but the iodine values were lowered and the refractive indices raised. In the second method examined the camphor was estimated polarimetrically at 20° C. in a half-shadow single-wedge compensating saccharimeter, a 200 mm. tube being used, and a 2 cm. cell of 10 per cent. dichromate solution interposed between the light and illuminating lens in order to compensate for difference in colours of the field. The calculated angular rotation for a 200 mm. tube was found to be 0.98 to 0.99 for each per cent. of camphor in the case of solutions of standard pharmacopœial strength; the oils alone showed slight optical activity, e.g.: Cotton-seed oil, -0.45° V; arachis oil, -0.1° V; olive oil, +0.2° V; and sesame oil, +2.85° V.

R. G. P.

New Test for Formaldehyde with Resorcinol and Sulphuric Acid R. Cohn. (*Chem. Zeit.*, 1921, 45, 997-998.)—The colour reactions of oxalic, formic, and tartaric acids, with resorcinol and sulphuric acid (*cf.* ANALYST, 1921, 296), are not sufficiently sensitive for the detection of these acids in preserved foodstuffs, though the reactions are not affected by acetic, lactic, succinic, citric, malic, and benzoic acids. The following test is given for formaldehyde: Two c.c. of a 1 per cent. aqueous solution of resorcinol are mixed with 2 c.c. of the liquid to be tested, and a layer of concentrated sulphuric acid is run underneath. A white flocculent precipitate, which gradually diffuses upwards, forms at the zone of contact, and under it is formed a violet-red ring. Oxalic and formic acids do not interfere, and give respectively a blue and orange ring, and if the sulphuric acid underneath is gently warmed a deep red colour is produced by tartaric acid, while the formalin white ring is not affected. The test is more sensitive than any previously described, and will detect 0.05 mgrm. of formaldehyde. The white flocculent ring is characteristic of aldehydes: formaldehyde gives a violet ring underneath, benzaldehyde gives a yellow ring, acetaldehyde a deep green, hexamine a greenish-brown, and furfuraldehyde gives a dark violet ring with a yellow layer below it. H. E. C.

Decomposition of Atropine. D. B. Dolt. (*Pharm. J.*, 1921, 107, 286-287.)—In the B.P. method of estimating alkaloids in belladonna extract, the final extraction with chloroform is made in presence of ammonia. Should only a slight excess of ammonia be added and the extraction be carried out at once, there will only be a slight loss of alkaloids, but if the solution be allowed to stand for some hours in contact with ammonia or sodium carbonate a large proportion of the alkaloids will be decomposed. For example, an extract of belladonna was diluted, acidified, and extracted with chloroform as the B.P. directs, and the acid aqueous portion divided into three parts, which were left for sixteen hours, one without addition, one with excess of sodium bicarbonate, and one with excess of ammonia. The amounts of alkaloids found in the three portions were 1.03, 0.91, and 0.52 per cent. respectively. Analogous results were obtained with solutions of pure atropine sulphate.

Salicin Content of British Columbian Willows and Poplars. R. H. Clark and K. B. Gillie. (*Amer. J. Pharm.*, 1921, 93, 618-620.)—The salicin content of the barks of a number of species of *Salix* and of *Populus* was estimated by a modification of the usual method of hydrolysis with emulsin. The following average amounts were found in the spring and autumn respectively: *Salix Nuttallii*, 3.90, 4.49; *S. Hookeriana*, 0.81, 5.09; *S. sitchensis*, 2.80, 7.38; *S. lasiandra*, 2.50, 2.51; *S. purpurea*, 3.83 (autumn); *Populus trichocarpa*, 0.96, 3.86; and *P. tremuloides*, 3.77, 2.45. per cent. R. G. P.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Occurrence of Quercetin in Brown-Husked Maize. C. E. Sando and H. H. Bartlett. (*J. Agric. Research*, 1921, 22, 1-4.)—The husks of a brown-husked type of maize contain free quercetin and a glucoside which, on hydrolysis, yields quercetin and dextrose. Both compounds are lemon-yellow in colour.

Chemical Study of Certain Pacific Coast Fishes. D. B. Dill. (*J. Biol. Chem.*, 1921, 48, 73-82.)—Analysis of the edible portion of twenty-one samples of the California mackerel (*Scomber japonica*) gave the following results: Total solids, 24.62 to 38.80; ether extract, 0.63 to 18.12; ash, 1.24 to 1.50; and nitrogen, 3.16 to 3.77 per cent. Large mackerel were usually fatter than small mackerel of the same school. Analysis of other species of mackerel-like fish are also given. The variations in the composition of mackerel during one season were not parallel to those of another season. With some exceptions, the fish showed an increasing fat content through the summer and early autumn.

Chemical Study of the California Sardine (*Sardinia caerulea*). D. B. Dill. (*J. Biol. Chem.*, 1921, 48, 93-103.)—Tables are given showing for the different months of the year the average composition of the edible portions of Californian sardines of different sizes, namely: (1) "Quarters," weighing 15 to 40 grms. each; (2) "halves," 48 to 70 grms.; (3) "small ovals," 80 to 120 grms.; and (4) "large ovals," 140 to 260 grms. For class (4) the percentage composition varies between the limits: Solids, 21.08 to 40.30; ether extract, 0.75 to 21.38; total nitrogen, 2.79 to 3.38. In three samples the percentages of glycogen were 0.50, 0.17, and 0.22 in the edible portion. The conclusions drawn from the results obtained are as follows: Individual sardines of the same size and from the same school vary greatly in composition; the fat content is at a maximum in the summer months with small sardines, and usually increases with the size of the fish; with large sardines the fat drops from a maximum in December or earlier to a minimum in May; there is no evidence that the growth of the reproductive organs draws largely on the reserve store of fat; any dependence of the percentage of fat in the sardine on the sea temperature is a remote one.

T. H. P.

Preservation of Fish Frozen in Chilled Brine. Penetration of Salt. L. H. Almy and E. Field. (*J. Ind. Eng. Chem.*, 1921, 13, 927-930.)—When fish (herrings, flounders, whiting, etc.) were frozen by immersion in chilled brine, the outer muscular layer, one-eighth inch in depth, absorbed from 0.32 to 6.22 per cent. of salt, calculated on the dry substance. The absorption of the salt was not affected by the concentration of the brine, or by slight alterations in the temperature of the latter. The greater part of the salt was absorbed during the first thirty minutes of the immersion period (two hours); when the fish was cooled to about 0° C. before immersion, the amount of salt absorbed increased considerably.

W. P. S.

Digestibility of Some Hydrogenated Oils. A. D. Holmes and H. J. Deuel. (*Amer. J. Physiol.*, 1921, 54, 479-488; *Chem. Abstr.*, 1921, 15, 2113.)—The digestibility coefficients for man of oils hydrogenated to different degrees of hardness were determined with the following results: Cotton-seed oil hydrogenated to melt at 35°, 38.6°, and 46° C., showed digestibility coefficients of 96.8, 95.5, and 94.9 per cent. respectively. In the case of hydrogenated arachis oil, melting at 37°, 39°, 43°, 50°, and 52.4° C., the respective digestibility coefficients were 98.1, 95.9, 96.6, 92.0, and 79.0 per cent. Hardened maize oil, melting at 33°, 43°, and 50° C., showed coefficients of 94.7, 95.4, and 88.5 per cent. respectively.

Examination of Proteins in Cow and Ox Serum, Colostrum, and Milk by Racemisation. H. E. Woodman. (*Biochem. J.*, 1921, 15, 187-201.)—The identity or non-identity of proteins was determined by the following method: The total globulin was removed by half-saturation of the neutral solution with ammonium sulphate, the pseudoglobulin and euglobulin being then separated by dialysis. The albumin in the filtrate from the total globulin was precipitated by the addition of the necessary amount of $\frac{N}{2}$ sulphuric acid and an equal volume of saturated ammonium sulphate solution, and the precipitate was freed from ammonium sulphate by dialysis. The proteins were finely ground and dried for several days *in vacuo* over calcium chloride, after which 1 grm. of the dry material was transferred to a 50 c.c. flask containing a little water, gently shaken to dissolve or keep the protein in suspension, and 25 c.c. of *N*-sodium hydroxide solution, free from carbonate, were run in. The flask was filled almost to the mark with water, and placed in an incubator at 37° C., and, on reaching that temperature, the liquid was made up to the mark, mixed, and filtered into a small stoppered flask, which was kept in the incubator. The optical rotation was read in a 100 mm. tube at intervals during about three hundred hours, the reading being then practically constant. A curve was plotted from the results, the abscissæ representing the number of hours and the ordinates the values of $[\alpha]_D$ obtained. Each protein showed a distinctive curve, giving the same initial and final rotations and the same rate of diminution throughout. Further, the same optical behaviour was observed when the proportions of alkali and protein were varied. The results show that euglobulin and pseudoglobulin are identical, whether derived from serum or colostrum; the albumin of milk is identical with that of colostrum; lactalbumin and serum albumin are distinct proteins; pseudoglobulin, whether coagulated or not, yields the same results; and globulin is a definite compound, the composition of which is unaffected by different methods of preparation. T. J. W.

Substances Precipitated by Mercuric Sulphate from Hydrolysed Caseinogen and the Isolation of Tryptophane. H. Onslow. (*Biochem. J.*, 1921, 15, 392-399.)—The following substances were isolated from the products of the hydrolysis of caseinogen with baryta by means of precipitation with mercuric sulphate, decomposition of the precipitate with hydrogen sulphide, further digestion with trypsin and erepsin, and reprecipitation of the products: Tyrosine, leucine, cystine, and other mono-amino acids, glutamic and aspartic acids, histidine and proline, but neither lysine nor arginine. Since some tyrosine remains combined after drastic acid hydrolysis of caseinogen, this amino-acid is not separated quantitatively early in digestion. By the following method a yield of 1.3 to 1.7 per cent. of tryptophane may be obtained without difficulty: One kilo of caseinogen is added gradually, with continual stirring, to a solution of 136 grms. of crystallised sodium acetate in 10 litres of tap-water. Strong sodium hydroxide solution is introduced until the suspension is alkaline to cresol red and acid to phenolphthalein, when the P_H value will be about 8.1. From 15 to 20 grms. of trypsin, 20 grms. of sodium fluoride, and 50 c.c. of toluene are then added, the mixture poured into large bottles, and placed in an incubator at a minimum temperature of 38° C. If, after three or four days, crystals of tyrosine have not formed on the sides of the bottles, more

enzyme is added, and the bottles returned to the incubator. On completion of digestion the mixture is filtered, after the addition of kieselguhr, and 13 c.c. of ice-cold 50 per cent. sulphuric acid are added to each 87 c.c. of the filtrate, followed by 120 to 150 c.c. of the mercuric sulphate reagent to every litre. The mixture is left overnight and then filtered, and the residue washed with 7 per cent. sulphuric acid until it gives a negative result with Millon's reagent. Finally, it is washed with water, then suspended in a litre of water, treated with hot 40 per cent. baryta solution until alkaline, and decomposed with hydrogen sulphide. The liquid is filtered, heated under reduced pressure to remove hydrogen sulphide, and treated with either baryta or sulphuric acid to adjust the P_H value to 8.1, when trypsin is again added and the digestion repeated during five to seven days. The resulting liquid is rendered neutral to litmus and concentrated under reduced pressure to about 170 c.c. The solution obtained is warmed to 70° C. and extracted with 10 to 15 portions of pure butyl alcohol, which is subsequently distilled under reduced pressure until tryptophane crystallises. After separation of the deposit, a second and third crop of crystals may be obtained by further concentration, until the solid is precipitated in a gummy condition. A yield of 10 to 15 grms. of tryptophane should be obtained, and it may be purified by boiling it with 60 per cent. alcohol and a little charcoal and recrystallising it. The gummy precipitate, which also contains tryptophane, may be added to a subsequent preparation before precipitation with mercuric sulphate.

T. J. W.

Action of Alkalis and of Pectase upon Pectin. F. Tutin. (*Biochem. J.*, 1921, 15, 494-497.)—Pectins prepared from apple pomace, carrots, turnips, and mangold wurzels, when purified, were immediately neutralised by 1.05 per cent. of their weight of sodium hydroxide, but after standing in the cold for half an hour in presence of excess of alkali it was found that they had taken up 12.35 per cent. of their weight of sodium hydroxide, this figure not being increased by prolonged standing. Addition of excess of acid produced a precipitate of pectic acid. In the same experiment repeated at 80° C. there was much decomposition, but there was no definite end point or precipitate of pectic acid. Where the cold mixture obtained in the first experiment was slightly acidified and fractionally distilled, acetone and methyl alcohol, in the respective proportion of 1 and 2, were the only products obtained. On treating a solution of pectin overnight with calcium carbonate and pectase (obtained by grinding clover with water), and subsequently distilling the liquid, acetone and methyl alcohol were obtained in the same proportion as by the action of alkali. The residue consisted of a gelatinous mass of calcium pectate. These results indicate that pectin probably consists of the dimethyl-isopropenyl ester of pectic acid.

T. J. W.

Supposed Importance of Vitamins in Promoting Bacterial Growth. J. W. McLeod and G. A. Wyon. (*J. Path. Bact.*, 1921, 24, 205-210; *Chem. Abstr.*, 1921, 15, 2291.)—Experiments were made with the object of utilising bacterial growth as an index of the presence of vitamins. The most striking results were obtained with a kidney extract, minute quantities of which had a stimulating action. The

growth-promoting power of fresh blood or serum did not show any definite relationship to the known vitamin content, whilst, on the other hand, charcoal, which is devoid of vitamins, had a stimulative effect. The growth-promoting power of a serum was partially destroyed by heat for the pneumococcus, but not for the meningococcus.

Seed of the Georgia Velvet Bean. B. Sure and J. W. Read. *J. Agric. Research*, 1921, **22**, 5-15.)—The Georgia velvet bean, *Stizolobium deeringianum*, is being cultivated in increasing quantities in the United States, and is usually ground up, either with or without the pods, for winter fodder. Experiments upon rats have shown that the bean is injurious if used to the extent of 40 per cent. of the ration. The injurious action is to a large extent eliminated by heating the seed for an hour in an autoclave, and up to 60 per cent. may then be used in the ration. Unlike most seeds hitherto studied, this bean is very abundant in the fat-soluble vitamin, which is not destroyed after the seed has been heated for an hour under a pressure of 15 lbs. The water-soluble vitamin, however, is of low concentration in the hulled seed. Two globulins and an albumin (low in histidin) have been isolated from the bean, but both the proteins and the salts in the seed are of a deficient character for growth.

Factors Influencing the Value of Milk and Butter as Sources of Vitamin A. J. C. Drummond, K. H. Coward and A. F. Watson. (*Biochem. J.*, 1921, **15**, 540-552.)—Rats whose growth had been inhibited by feeding them upon a diet deficient in vitamin A were supplied daily with 2 c.c. of milk or 0.2 grm. of butter, and, after this had been completely consumed, the ration of basal food mixture was supplied. Other groups of the animals were furnished with different quantities of milk and butter until a satisfactory rate of growth was obtained. The following deductions were made from an extensive series of experimental results: The vitamin content of butter is diminished if the food of the animal from which it is derived is deficient in vitamins. The milk obtained from different breeds of grass-fed cows varies, and there is frequently correlation between the amount of pigment stored in the body fat of the animal and the vitamin content of the milk. Colostrum possesses a higher proportion of vitamin than the later milk, an observation which should be of service in practical nutrition. Butter shows from one-third to one-half the vitamin activity of the milk from which it was derived. The loss is partly due to mechanical separation, and partly due to destruction. Storage of butter and the development of rancidity cause no diminution in the vitamin content, if oxidation, as indicated by bleaching of the natural pigment, is not allowed to occur. "Renovation" of rancid butter by blowing air through the melted material causes a rapid decrease in the vitamin content.

T. J. W.

Differential Dialysis of Vitamins. S. S. Zilva and M. Miura. (*Biochem. J.*, 1921, **15**, 422-426.)—Collodion thimbles of varying degrees of permeability were prepared by immersing them in different concentrations of alcohol for twenty-four hours. Their relative permeability was ascertained by suspending them in running water, introducing various salt and dye solutions, and observing the time and degree of diffusion.

Yeast autolysed at 37° C. was used as the source of the antineuritic vitamin B, whilst the antiscorbutic vitamin C was derived from lemon juice neutralised by the addition of excess of calcium carbonate. The vitamin-containing solutions were dialysed in thimbles of different permeability, and then tested on rats and guinea pigs, which were fed on a basal diet free from the vitamin under examination. It was found that neither of the vitamins tested would pass through thimbles treated with 90 per cent. alcohol, but completely diffused through one treated with 95 per cent. alcohol in four days. These results agree with those obtained when dialysing solutions of methylene blue, neutral red, and safranin, and the deduction is drawn that these vitamins may be of a semi-colloid nature, but there is no evidence to show whether the molecules are simple or associated.

T. J. W.

The "Bachman Test" for Water-Soluble Vitamin B. W. H. Eddy and H. C. Stevenson. (*Proc. Soc. Expt. Biol. Med.*, 1919, 17, 52-56; *Chem. Abstr.*, 1921, 11, 1745.)—Yeast cells are sown in Nageli's solution contained in fermentation tubes, and incubated to induce gas formation. This solution consists of 10 grms. of dextrose, 1 grm. of ammonium nitrate, 0.05 grm. of calcium phosphate, 0.5 grm. of potassium hydrogen phosphate, and 0.25 grm. of magnesium sulphate in 100 c.c. of water. Gas evolution proceeds very slowly, or not at all, but on the addition of extracts containing vitamin B the production of gas is accelerated. The authors have confirmed these results, and have found that extracts of navy beans, starch, alfalfa, protozoa food mixtures, protein-free milk, human milk, whole milk, and milk containing small amounts of sodium hydroxide, stimulate gas production in different degrees. It is suggested that both the detection of vitamin B and estimation of the vitamin content may be satisfactorily achieved by further standardisation of the test.

T. J. W.

Colorimetric Estimation of Carnosine. W. M. Clifford. (*Biochem. J.*, 1921, 15, 400-406.)—The precautions emphasised by Koessler and Hanke (*J. Biol. Chem.*, 1919, 39, 497) with regard to mixing the solutions in the colorimeter cup are unnecessary. Since the colour developed is not proportional to the amount of carnosine present there is no linear relation between the two. The following modification of those authors' method is recommended: A weighed quantity of the finely minced tissue is extracted for one hour with a known volume of water at 90° C. The liquid is then filtered, and the residue squeezed out; from 2 to 10 c.c. of 20 per cent. metaphosphoric acid are added to the extract, and the mixture allowed to stand for two to twenty-four hours. After filtration, a definite volume is transferred to a graduated flask, rendered neutral to litmus by the addition of 10 per cent. sodium hydroxide solution, and diluted to a known volume with distilled water. Ten c.c. of 1.1 per cent. sodium carbonate solution are diluted with $2-x$ c.c. of water, and 4 c.c. of Koessler and Hanke's diazobenzenesulphanilic acid reagent added. Exactly one minute later x c.c. of the extract are added, and the solution is transferred to one of the colorimeter cups adjusted to a thickness of 20 mm. On the solution developing its maximum colour intensity, which occurs about five minutes after mixing, it is compared with the Congo-red methyl orange standard employed by the previous

authors. The estimation is repeated until 20 mm. of the unknown solution matches 30 mm. of the standard, when the volume of extract taken contains 0.1 mgrm. of carnosine. Parallel estimations agree within 0.05 mm. of the colorimeter scale.

T. J. W.

Methods for the Estimation of Cholesterol and Allied Substances. J. A. Gardner and M. Williams. (*Biochem. J.*, 1921, 15, 363-375.)—In the application of Windaus's method (*ANALYST*, 1910, 35, 256) it is necessary, in order to obtain accurate results, to add from 1.5 to 2.5 per cent. excess of digitonin, and to make allowance for the solubility of the digitonides in alcohol. Thus the solubility of the cholesterol compound in 95 per cent. alcohol is 0.014 and 0.16 per cent. at 18° and 72° C. respectively. Coprosterol digitonide is more soluble than other members of the group, 96 per cent. alcohol dissolving 0.143, 0.24, and 0.83 per cent. at 10°, 15°, and 78° C. respectively. A very slight error is introduced by the washing with ether or petroleum spirit, but is in most cases negligible. Investigation of the colorimetric method, in which acetic anhydride and sulphuric acid are used, showed that the same volume of acid should be used throughout a series of estimations, approximately the same temperature should be maintained, and that varying amounts of acetic anhydride had little effect upon the result when this substance was in large excess. The digitonin method yields accurate results with ether extracts of tissues and with animal fluids, whilst the colorimetric method (which is applicable to smaller quantities) is satisfactory with serum and blood, but is more or less unreliable with tissue extracts, owing to variation in the colours obtained.

T. J. W.

Source of Error in the Colorimetric Estimation of Cholesterol in Tissue Fats. J. A. Gardner and F. W. Fox. (*Biochem. J.*, 1921, 15, 376-378.)—Ether extracted from alcoholic potassium hydroxide solutions, which had been heated and then diluted with water, a small amount of a resinous substance, which on solution in chloroform and the addition of acetic anhydride and sulphuric acid produced a brown or red coloration, which on standing changed to a yellowish-green tint. Similar results were obtained with the duty-free non-methylated alcohol, supplied to laboratories, when boiled with sodium hydroxide or heated in sealed tubes to 110° C., or in which sodium had been dissolved, but the brown coloration did not in all cases become green. Unsuccessful attempts were made to purify the alcohol by heating with sodium hydroxide and subsequent distillation and by other methods. The alcohol and ether used, when evaporated to dryness without the addition of alkali did not give this reaction. Since the hydrolysis of fats for the estimation of sterols is effected by the use of alcoholic potassium hydroxide or sodium ethoxide solution, the coloration due to the above reaction is liable to vitiate results obtained when the acetic anhydride-sulphuric acid method is adopted.

T. J. W.

Estimation of Sugar in Normal Urine. S. R. Benedict and E. Osterberg. (*J. Biol. Chem.*, 1921, 43, 51-57.)—The great labour involved in the mercuric nitrate method for estimating sugar in urine (*J. Biol. Chem.*, 1918, 34, 195) is avoided by the following procedure: The urine is diluted to the sp. gr. 1.025 to 1.030, and 15 c.c.

mixed with about 1 gm. of bone black (see below) and the mixture occasionally shaken vigorously during five to ten minutes, and then filtered through a small dry filter. Such volume of the filtrate as contains about 0.001 gm. of sugar is measured into a large test-tube with a mark at 25 c.c., and water added, where necessary, to bring the volume to exactly 3 c.c. Exactly 1 c.c. of 0.6 per cent. picric acid solution (best prepared from dry picric acid) and 0.5 c.c. of 5 per cent. sodium hydroxide solution are added, and 5 drops of 50 per cent. aqueous acetone (prepared fresh every day or two) are dropped directly into the liquid, not on to the side of the tube, and the tube shaken gently and immediately immersed in boiling water for twelve to fifteen minutes. Similar procedure is followed with 3 c.c. of pure dextrose solution containing 0.001 gm. of the sugar, the two tubes being heated simultaneously. Colorimetric comparison of the two liquids is then made. When a 0.001 gm. standard is used, satisfactory results may be obtained with solutions containing between 0.00075 and 0.00175 gm. of sugar; if less than 0.0007 gm. of sugar is to be estimated it is preferable to use a standard solution containing 0.0005 gm. in 3 c.c., and to dilute in both cases to 12.5 instead of 25 c.c. The bone black is prepared by boiling 250 grms. of the ordinary commercial product with about 1.5 litres of hydrochloric acid (1 vol. acid and 4 vols. water) for about thirty minutes, collecting it on a large Buchner funnel, washing it with hot water until the washings are neutral to litmus, drying, and powdering it. For clinical purposes the use of bone black may be omitted, the resultant figures being then about 0.03 to 0.04 per cent. too high. The new method yields results which are slightly lower than those given by the mercuric nitrate method. T. H. P.

Rapid Method for the Estimation of Hippuric Acid in Urine. F. B. Kingsbury and W. W. Swanson. (*J. Biol. Chem.*, 1921, 48, 13-20.)—Fifty c.c. of the urine are boiled in a 500 to 800 c.c. Kjeldahl flask with 7.5 grms. of sodium hydroxide and 0.5 gm. of magnesium oxide, so that the volume is reduced to about 25 c.c. in 30 minutes. To the boiling liquid 1 c.c. of 7 per cent. potassium permanganate solution (approximately saturated at room temperature) is added, any left on the neck being washed down with as little water as possible. The flask is twirled gently for a minute or two, cooled under the tap, and fitted with a fairly tight test-tube condenser, down the outside of which 30 c.c. of concentrated nitric acid are slowly poured. The mixture, which is cleared by the acid, is now gently boiled for thirty minutes, or, if a lighter extract easier to titrate is desired, for forty-five minutes, with a good flow of water through the condenser, the flask being afterwards cooled under the tap, the condenser rinsed down with 25 c.c. of water to remove any benzoic acid sublimed on the bottom of the condenser, and the contents of the flask transferred to a 500 c.c. separating funnel containing 25 grms. of solid ammonium sulphate; the flask is rinsed with 20 c.c. of water into the funnel. When the ammonium sulphate is dissolved, the benzoic acid is extracted successively with 50, 35, 25, and 25 c.c. portions of neutral, well-washed chloroform, the first two portions being used to rinse the Kjeldahl flask. The combined extracts are washed in a second separating funnel with 100 c.c. of a solution of 1 c.c. of concentrated hydrochloric acid in 2 litres of saturated sodium chloride solution and drawn off through a dry filter-paper into a dry Erlenmeyer flask. The separating funnel from which

the extract has been drawn is rinsed with 20 c.c. of chloroform, this being transferred to a small beaker containing the wet filter-paper, which is then rinsed with the chloroform, the latter being poured through a dry filter into the Erlenmeyer flask containing the bulk of the extract. This is then titrated in presence of 4 drops of a 1 per cent. solution of phenolphthalein in absolute alcohol, with $\frac{N}{10}$ sodium ethoxide solution (cf. Folin and Flanders, *J. Biol. Chem.*, 1912, 11, 257) until a faint but distinct pink coloration is obtained. When, as in the case of nephritic urine, protein is present, the urine is collected in about 15 c.c. of 2 per cent. nitric acid. Fifty c.c. of the urine, treated with 3 or 4 drops of 0.1 per cent. solution of methyl red in alcohol, are rendered just distinctly yellow by the addition of approximately *N*-sodium hydroxide solution. The liquid is then boiled and, while boiling, is treated with sufficient $\frac{N}{10}$ hydrochloric acid to give it the first definite red colour. This procedure removes the albumin almost quantitatively, so that the subsequent titration is not affected. The coagulum of albumin on the filter-paper is washed twice with 50 c.c. of boiling water, the combined filtrate and washings being rendered slightly alkaline to methyl red, and evaporated rapidly in an 800 c.c. Kjeldahl flask over a naked flame, any bumping or frothing being checked by adding a glass bead and a drop of capryl alcohol. By supporting the funnel in the neck of the flask by means of a slice of a large cork the filtration and evaporation may be effected simultaneously. When the volume of the liquid has been reduced to about 50 c.c., 7.5 grms. of sodium hydroxide and 0.5 gm. of magnesium oxide are added and the procedure described above then followed. The method requires about two hours or, with albuminous urine, about three hours, and gives results agreeing well with those obtained by the longer Folin and Flanders' method.

T. H. P.

Estimation of Creatinine in the Presence of Acetone and Diacetic Acid.
N. F. Blau. (*J. Biol. Chem.*, 1921, 48, 105-118.)—Conflicting statements have been published regarding the effects of acetone and acetoacetic acid on the colorimetric estimation of creatinine in urine by means of alkaline picrate. The author finds that the coloration obtained is lessened in intensity by acetone in large proportion, and that the upper limit of permissible acetone may be set at 0.5 per cent.; with no amount of acetone was deepening of the colour produced. In small proportions ethyl acetoacetate lessens, and in larger proportions enhances, the intensity of the coloration, the position of the line of demarcation being difficult to establish. Acetoacetic acid, even in small amount, has a marked influence on the result, and the following method has now been elaborated for the preliminary removal of this acid from urine. If the urine is alkaline, it is treated with concentrated hydrochloric acid until its reaction is that of weak organic acids—that is, red to litmus, but not blue to Congo-red. Ten c.c. of the urine and 5 volumes of methyl alcohol are introduced into a 300 c.c. Erlenmeyer or round-bottomed flask containing a few glass beads, and the mixture slowly boiled over an asbestos mat until one to two minutes after the temperature has risen to 100° C. (In this operation, which should occupy not less than fifteen minutes, the methyl alcohol may be distilled through a condenser, the recovered alcohol being readily purified by heating in a reflux apparatus with mercuric sulphate, filtering, and fractionally distilling the filtrate.) The flask is

thoroughly cooled in running water, its contents then being mixed with 15 c.c. of saturated picric acid solution and 5 c.c. of 10 per cent. sodium hydroxide solution, and, after the lapse of eight minutes, washed quantitatively into a 500 c.c. flask, diluted to the mark, and the coloration read. This method is found to give accurate results.

T. H. P.

WATER ANALYSIS.

Fluorescein as an Aid to Tracing Underground Water. H. Stabler. (*Eng. News-Record*, 1921, 86, 639-640; *Chem. Abstr.*, 1921, 12, 1956.)—A solution of fluorescein containing 1 part in 40,000,000 is recognised by the naked eye, but practical experience has shown that the quantity used should be calculated to produce about 1 part in 10,000,000. Successful use has been made of this method in tracing underground connections, detecting leakages in canals, etc. T. J. W.

Estimation of Phosphates in Water. D. Florentin. (*Ann. Chim. anal.*, 1921, 3, 295-296.)—Denigès' test (*ANALYST*, 1921, 24) for phosphates or arsenates is modified so as to furnish a colorimetric method for the estimation of phosphates in water: Ten c.c. of the water are treated with 3 to 4 drops of an acid solution of ammonium molybdate (100 c.c. of 10 per cent. ammonium molybdate, 300 c.c. of 50 per cent. [by weight] sulphuric acid) and then with 1 to 4 drops of stannous chloride solution (0.1 gram. of tin per 10 c.c.). In the presence of phosphates a blue colour appears in a few seconds, becomes more intense, and is constant after ten minutes: 0.01 mgrm. P_2O_5 per litre can thus be detected. The colour obtained is compared with those of standards prepared with known amounts of phosphate, but, as such standards are not stable, permanent standards prepared from indigo-carmin are convenient. Silicates also produce a blue coloration in this test, but as 0.1 gram. of sodium silicate per litre gave no appreciable colour, silicates likely to be present in water would have no serious influence.

B. G. P.

AGRICULTURAL ANALYSIS.

Method of Measuring Soil Toxicity, Acidity, and Basicity. R. H. Carr. (*J. Ind. Eng. Chem.*, 1921, 13, 931-933.)—In an acid soil a portion of the iron and aluminium is present in a readily soluble form, and can be extracted by salt solutions which are nearly neutral; if potassium thiocyanate be used, the coloured ferric thiocyanate solution is decolorised by the addition of a base, the change occurring at a hydrogen ion concentration of $P_H = 5.5$. Fifty grms. of the soil are shaken in a stoppered cylinder with 30 c.c. of a saturated solution of potassium thiocyanate in 95 per cent. alcohol (adjusted to $P_H = 5.3$). If the liquid is pink after the soil has settled, $\frac{N}{10}$ alcoholic potassium hydroxide solution is added until the pink coloration disappears. The mixture is then placed aside overnight and again titrated, if necessary. Each c.c. of $\frac{N}{10}$ alkali solution used is equivalent to 200 lbs. of calcium carbonate per acre. In cases where the pink colour does not appear when the soil is shaken with thiocyanate solution, the mixture is titrated with $\frac{N}{10}$ alcoholic hydrochloric acid solution; the calcium carbonate equivalent is calculated from the

amount of acid used. The relative amount of aluminium in solution is indicated by the depth of the blue coloration obtained when the potassium thiocyanate extract of the soil is treated with a few drops of logwood tincture. Estimation of the quantity of soluble iron and aluminium compounds is helpful in pointing to a source of some of the plant disease troubles.

W. P. S.

ORGANIC ANALYSIS.

Estimation of Alcohols and Phenols in Essential Oils by Esterification with Pyridine. H. W. van Urk. (*Pharm. Weekblad*, 1921, 58, 1265-1269).—In the method of Verley and Bolsing (*ANALYST*, 1902, 27, 60) the alcohol is heated on the water-bath with excess of a solution of acetic anhydride in pyridine, water added to the cooled mixture, and the uncombined acetic acid titrated. A systematic examination of this method under varying conditions with santal wood, peppermint, and clove oils has shown that the results obtained by the addition of 50 c.c. of the reagent are higher than with the specified 25 c.c., although still too low. For example, the amount of menthol in a peppermint oil ranged from 34.5 to 40.6 per cent.; that of santalol in santal-wood oil from 87.76 to 91 per cent. (92.3 by Thoms' method); and that of eugenol in clove oil from 71.6 to 72.8 (74.8 per cent. by Thoms' method). Hence the method is only suitable for the approximate analysis of the two latter oils, and is unsuitable for peppermint oil.

Estimation of Aceto-acetic Ester. H. Yanagisawa and M. Kamio. (*J. Pharm. Soc., Japan*, 1921, 240-246; *Chem. Abstr.*, 1921, 15, 2261).—Ten grms. of the sample are dissolved in 100 c.c. of dilute alcohol, and 5 c.c. of the solution are treated with 50 c.c. of an aqueous solution of recrystallised sodium sulphite (25 grms. in 100 c.c.), and titrated with *N*-sulphuric acid with phenolphthalein as indicator. A separate estimation of the free acid present is made by diluting 5 c.c. of the solution with 20 c.c. of water and titrating it with standard alkali, and also of the amount of standard sulphuric acid required to give an acid reaction with 50 c.c. of the sulphite solution, and corrections are applied to the results of the first titration. The method, which is not applicable in the presence of any other aldehyde or ketone, depends upon the fact that 3 mols. of aceto-acetic ester react with sodium sulphite, liberating 2 mols. of sodium hydroxide.

Volumetric Estimation of Phenylhydrazine and Its Application to the Estimation of Pentosans and Pentoses. A. R. Ling and D. R. Nanji. (*Biochem. J.*, 1921, 15, 466-468).—The addition of four or more equivalents of iodine to one of phenylhydrazine produces iodobenzene, hydriodic acid and nitrogen. Upon this reaction the authors base the following method of estimating pentosans and pentoses. A known weight of the substance is distilled with 12 per cent. hydrochloric acid until the distillate ceases to yield a pink colour with aniline acetate paper, when the whole distillate is diluted to 250 c.c. Twenty-five c.c. of this solution are titrated with 3-*N* sodium hydroxide solution in a 100 c.c. flask until neutral to methyl orange, an increase of temperature being avoided. The solution is slightly acidified with acetic acid, treated with 10 c.c. of an approximately 2 per cent.

aqueous solution of phenylhydrazine, diluted with water to reach the neck of the flask, which is then closed with a cork and heated for 20 minutes at 50° to 55° C. On cooling, the solution is diluted to 100 c.c. and filtered through a hardened filter-paper. Ten c.c. of the filtrate are run into 10 c.c. of $\frac{N}{10}$ iodine solution, and the mixture diluted to about 100 c.c., and titrated with $\frac{N}{20}$ sodium thiosulphate solution. A similar estimation is made with the original phenylhydrazine solution, and the difference between the amounts of sodium thiosulphate required in the two estimations is equivalent to the furfural present. The results quoted for arabinose agree well with those given by the phloroglucinol method. Sulphuric acid cannot be used in place of hydrochloric acid for the furfural distillation; when an equivalent strength of the former was used and the distillation extended over eight hours, only 85 per cent. of the possible furfural was obtained.

T. J. W.

Action of Anhydrous Stannic Chloride on Petroleum and Petroleum Distillates. G. Torossian. (*J. Ind. Eng. Chem.*, 1921, 13, 903-904.)—When anhydrous stannic chloride is added to crude petroleum or any of its distillates, a precipitate is formed immediately; the quantity, colour, and characteristics of the precipitate depend on the nature of the petroleum. The precipitate is separated readily by filtration, and, if an excess of stannic chloride has been used, the filtrate does not yield any further quantity of precipitate when more of the reagent is added. The stannic chloride reacts primarily with oxygenated bituminous substances, sulphur compounds, unsaturated organic acids and terpenes present in crude petroleum. The treatment decreases the colour of the petroleum product, but a subsequent treatment with an alkali solution is required to remove acidity, since stannic chloride does not eliminate the naphthenic acids completely.

W. P. S.

Effect of Variation in the Analytical Constants of Linseed Oil and Soya Bean Oil on the Estimation of these Oils by Means of the Hexabromide and Iodine Values. E. A. Tschudy. (*J. Ind. Eng. Chem.*, 1921, 13, 941-943.)—The hexabromide method gives results differing by +7 to -3 per cent. from the actual amount of linseed oil in mixtures of linseed oil and soya bean oil containing oils having the widest range of analytical constants, when average constants are used as a basis for the calculation; including the experimental error of the hexabromide method, the results obtained may differ by +13 to -9 per cent. from the true figure. If the amount of linseed oil in such a mixture be calculated from the iodine value, the result may differ by +16 to -17 per cent. from the quantity actually present.

W. P. S.

Need of a New Table for Specific Gravity and Percentage of Glycerol. L. W. Bosart. (*J. Ind. Eng. Chem.*, 1921, 13, 944-945.)—Gerlach's table, showing the percentage of glycerol corresponding with specific gravities at 15° C. is the most accurate one yet published, and is trustworthy, especially at the higher concentrations; his table of specific gravities at 20° C. is, however, inaccurate, and should be discarded. It is very desirable that new tables should be prepared, showing the specific gravities of glycerol solutions at 15.5° C. and 25° C.; until this is done the

following rule may be adopted: For 15° C. use Gerlach's table for that temperature; for 15.5° C., deduct 0.0002 from the specific gravity shown in Gerlach's table for 15° C., and for 25° C. use Gerlach's table for 20° C.

W. P. S.

Rapid Method for the Estimation of Fermentation Glycerol. K. Fleischer. (*Zeitsch. anal. Chem.*, 1921, 60, 330-338.)—The oxidation and the iodimetric methods being unsuitable for the estimation of the glycerol in the liquor from the sugar fermentation process of manufacture, the following rapid method of fractional distillation under reduced pressure has been perfected. A steam generator is connected, through a spiral copper superheater (with a thermometer) with a litre Classen flask fitted with a dropping funnel; the flask stands in a hot-air bath, and its side tube is connected with a round-bottomed flask in a water-bath. Into the latter flask passes a capillary tube, through which tiny bubbles of air are slowly admitted, and with it is connected a reflux condenser in which circulates warm water from the water-bath. The top of the reflux condenser is connected with an ordinary cold water Liebig condenser, to the receiver of which is attached the vacuum pump. For the estimation, steam superheated to 150° C. is introduced, 100 c.c. of the liquor are slowly dropped in from the funnel, and the air-bath gradually heated to 250° C. The water-bath is maintained at 70° C., and the pressure at 15 to 20 mm. When all the glycerol has distilled into the round-bottomed flask the steam supply is cut off, and the flame removed from under the air-bath; then the temperature of the water-bath is raised to the boiling-point, and the circulating water in the reflux condenser is also heated nearly to 100° C. The estimation is completed when there is no further distillation of water from the glycerol in the round flask. The glycerol is then weighed and its density determined by means of a pycnometer, from which data the percentage in the liquor is calculated. There is a constant small error, which, for the apparatus described, was found to be 1 per cent.; this figure is therefore added to the quantity found by experiment, and the result so obtained is within about 1 per cent. of the truth.

H. E. C.

Colour Reaction of Glycine Anhydride and of its Dipeptide Compounds. T. Sasaki. (*Biochem. Zeitsch.*, 1921, 114, 63-66; *Chem. Abstr.*, 1921, 12, 1914.)—These compounds yield red colorations with picric acid and sodium carbonate, similar to that produced by the Jaffé reaction for creatinine, but less intense. The reaction is not due to the presence of nitrogen in the molecule, since a similar coloration is produced by sugars. The author considers the active constituent to be a labile hydrogen atom, but is unable to decide upon which configuration of the molecule the colour development depends.

T. J. W.

Estimation of Sulphide, Sulphate, and Organic Sulphur in Coal. Alfred R. Powell. (*Techn. Paper 254, U.S. Bureau of Mines*, 1921, pp. 21.)—Test analyses carried out on various kinds of coals proved that Powell and Parr's method (*Bull. 111, Eng. Experimental Station, University of Illinois*, 1919, 10-35) gives accurate results. The air-dried coal is crushed to pass a 100-mesh screen. *Sulphate sulphur*.—Five grms. are digested at 60° C. for forty hours with 300 c.c. of 3 per cent.

hydrochloric acid, the filtered liquid boiled with a little bromine water and treated with ammonia. The iron in the precipitate is estimated volumetrically, and the sulphur trioxide in the filtrate as barium sulphate. *Sulphide sulphur*.—One grm. is digested at ordinary temperature for twenty-four hours with 80 c.c. of nitric acid (1 : 3 water; sp. gr. 1.12), with occasional stirring. The filtered liquid is evaporated to dryness with 2 c.c. of strong hydrochloric acid on a water-bath, the residue dissolved in dilute hydrochloric acid, and the solution poured into excess of hot dilute ammonia solution. The iron in the precipitate and the sulphur trioxide in the filtrate are estimated as before. The amount of sulphur thus found, less that of the sulphate sulphur, gives the percentage of pyritic sulphur; similarly, the iron present in pyrites is found by subtracting the first from the second iron figure. The pyritic sulphur, as calculated from the iron figure, should agree closely with the percentage found by assay. It is even recommended, for commercial work, to calculate the pyritic sulphur from the pyritic iron value, because the estimation is quicker and more accurate, and dilute nitric acid will dissolve small amounts of organic sulphur from certain coals. *Organic sulphur*.—In commercial work this is taken as the difference between the total sulphur (estimated by Eschka's method) and the sulphur present as sulphide and sulphate. The organic sulphur is present in two forms—viz., phenol-soluble and humus-sulphur. *Phenol-soluble sulphur* is estimated by digesting 1 grm. of coal for twenty hours at 150° C., under a reflux condenser with 25 c.c. of pure phenol, filtering while hot through a Gooch crucible, transferring the whole of the residue to the crucible with alcohol, and washing until the alcohol becomes colourless. The residue is washed with ether, dried in an oven, and ground up, together with the asbestos mat, with two parts of Eschka mixture; the mixture is ignited and the estimation finished as usual; the amount found, subtracted from the total sulphur, gives phenol-soluble sulphur. The *total organic sulphur* may be estimated directly by digesting the residue from the dilute nitric acid extraction, together with the filter-paper, with strong nitric acid on a water-bath until the acid has evaporated. With semi-bituminous coals, the acid treatment should be repeated several times. The dry residue is stirred up with 25 c.c. of strong ammonia and the liquid diluted and filtered. The filtrate is evaporated to dryness on a water-bath, and the residue ground up with Eschka mixture, etc., as usual. W. R. S.

INORGANIC ANALYSIS.

Methyl Orange as an Indicator in Presence of Indigo Carmine. F. X. Moerk. (*Amer. J. Pharm.*, 1921, 93, 675-679).—A mixture of methyl orange and indigo carmine furnishes a more sensitive indication of the end point in many acid and alkali titrations than methyl orange alone, and particularly in the titration of yellow-coloured solutions (Luther, *Chem. Zeit.*, 1907, 960). The author recommends the use of an indicator composed of methyl orange 1.0 grm., indigo carmine 2.5-3.0 grms., water 1 litre. Two portions of 0.2 c.c. of this solution are each diluted with 100 c.c. of water, and to the green or greenish-yellow solution sufficient $\frac{1}{10}$ acid is added to produce a "neutral tint"—i.e., to discharge the yellow or green colour without producing a red or violet colour. One portion is reserved as a

standard, and the substance to be tested is added to the other portion, which will turn violet or purplish with acids, and yellow or green with alkalis; this liquid is then titrated with standard acid or alkali until the colour matches the standard "neutral tint."

R. G. P.

Preparation of Standard Alkali Solutions Free from Carbonate. I. M. Kolthoff. (*Pharm. Weekblad*, 1921, 58, 1413-1419.)—A simple method of preparing sodium hydroxide solutions free from carbonate has been based on the observations of Anselme (*Bull. Soc. Chim.*, 1875, 24, 192), that the solubility of calcium oxide in $\frac{N}{2}$ sodium hydroxide solution at 20° C. is 20 mgrms. per litre, as compared with a solubility of 1,170 mgrms. in pure water. A litre of a 1.1 *N*-solution of commercial sodium hydroxide is shaken repeatedly with 50 c.c. of milk of lime, the mixture allowed to stand for at least a day, and the clear liquid drawn off, with precautions to prevent contact with atmospheric carbon dioxide. When diluted to $\frac{N}{10}$ strength the solution will contain, at most, 1 to 2 mgrms. of calcium per litre.

Use of Sodium Persulphate in Analysis. L. Débourdeaux. (*Bull. Sci. pharmacol.*, 1921, 28, 145-155, 191-192; *Chem. Abstr.*, 1921, 15, 2047, 2260.)—Finely divided silver is converted by sodium persulphate in acid solution into a compound described as "silver peroxide." This compound is immediately reduced in the presence of oxidisable substances and is then reoxidised, and this transference of oxygen continues until the reaction is complete, this being indicated by a permanent brown coloration attributed to the silver peroxide. This latter compound, however, continues its action so long as any sodium persulphate is present. The stability of the persulphate with respect to changes of temperature is greater in non-catalysed alkaline solutions than in the preceding case, and the reaction is complete when the solution is colourless; it then shows no charring on evaporation and calcination of the residue, and does not reduce potassium permanganate. The analysis of complex compounds not readily oxidisable by the usual means may be effected by successive oxidation in non-catalysed alkaline solution and in silver-catalysed acid solution. Sodium persulphate may be used for the estimation of carbon in the following classes of compounds: (1) Compounds—*e.g.*, acetic or oxalic acids, which yield carbon dioxide when treated with the reagent in acid solution; (2) those which are completely oxidised in an alkaline solution of the persulphate—*e.g.*, cacodylic acid; (3) compounds soluble in water and acids, but which, on combustion, yield incompletely oxidised products—*e.g.*, lactic acid; (4) compounds yielding insoluble products—*e.g.*, acetyl-salicylic acid; and (5) compounds insoluble in acid, but soluble in alkaline solutions—*e.g.*, terpin, camphoric acid, etc.

Estimation of Minute Amounts of Bromine. E. Oppenheimer. (*Arch. Exp. Path. Pharm.*, 1921, 89, 17-28; *Chem. Abstr.*, 1921, 15, 2045.)—A standardised chlorine solution, prepared from chloride of lime, is added in increasing quantities, in the presence of magenta and sulphuric acid, to the unknown solution and to solutions containing definite amounts of bromine. Bromo-fuchsin is formed by the

liberated bromine, and the amount of the latter is estimated colorimetrically by comparison of the changes of colour from violet and yellow.

Estimation of Sulphur Dioxide and Trioxide in Sulphur Burner Gases.

R. Dieckmann. (*Papierfabr.*, 1921, 19, 285-287; *Chem. Abstr.*, 1921, 15, 2261).—The method depends upon the use of methyl orange and phenolphthalein as joint indicators. The gases are passed into an Orsat apparatus, and the first change of colour, from red to yellow, gives the volume of both sulphur dioxide and trioxide; the passage of the gas is then continued until the sodium hydroxide is neutralised, and the difference between the two readings gives the amount of sulphur dioxide.

Estimation of Nitrous Fumes in Air. **J. Moir.** (*J. S. African Assoc.*

Anal. Chem., 1921, 4, 3-7; *Chem. Abstr.*, 1921, 15, 2044.)—A new test for nitrites or nitrous acid is described, which is similar to that of Ilosvay, but in which *p*-nitroaniline and α -naphthol are used instead of sulphanilic acid and naphthylamine. Two solutions are required: (a) consists of a solution of 1.5 grms. of *p*-nitroaniline in 40 c.c. of hydrochloric acid, diluted to 500 c.c.; (b) contains 2 grms. α -naphthol, 1.1 grm. of sodium hydroxide, and 10 grms. of sodium acetate per 500 c.c. The liquid to be tested for traces of nitrite is mixed with 1 to 10 c.c. of (a), heated to 50° C. until diazotisation is complete, after which solution (b) is added in the same quantity as (a). In the presence of a nitrite an orange-red precipitate of *p*-nitrobenzeneazo- α -naphthol is quantitatively obtained. It is collected, after an hour, in a Gooch crucible, dried, and weighed, 293 parts being equivalent to 92 parts of NO₂ or 69 parts of NaNO₂. The dyestuff dissolves in sodium hydroxide solution, producing an intense bluish-violet coloration, which may be used for a colorimetric estimation. *Fuse Igniter Fumes.*—According to the British Government requirement the fumes from 100 grms. of fuse must not contain more than 0.5 grm. of nitrogen peroxide. To distinguish between this compound and nitrite in smoke use is made of the acidity of the former. The following methods have given satisfactory results: (1) The gas is kept in contact with neutral hydrogen peroxide solution for twenty-four hours, and the solution then titrated with $\frac{N}{10}$ sodium hydroxide solution, with methyl orange as indicator ($\text{NO}_2 + \text{H}_2\text{O}_2 = 2\text{HNO}_3$). It was found as an empirical factor that 1 c.c. of the standard alkali = 0.0060 grm. NO₂. (2) The fumes are treated with a solution of potassium iodide (1 grm. in 20 c.c.) for at least six hours with occasional shaking, during which the walls of the container should be thoroughly wetted, and the liberated iodine is titrated with $\frac{N}{10}$ thiosulphate solution. One c.c. of the latter is equivalent to 0.0051 grm. of nitrogen peroxide, instead of the theoretical value.

New Method for the Estimation of Arsenic. **J. Cribier.** (*J. Pharm.*

Chim., 1921, [vii], 24, 241-246.)—The yellow coloration produced by hydrogen arsenide on mercuric chloride paper is converted into deep brown when the paper is immersed in 10 per cent. potassium iodide solution, and after the paper has been washed and dried this coloration exhibits great stability towards light and moisture. The yellow or grey colorations produced by hydrogen sulphide, phosphide,

or arsenide with mercuric chloride are also fixed by potassium iodide, but the original colour is not changed. Further, compounds capable of yielding hydrogen sulphide or phosphide, when reduced by means of zinc and sulphuric acid, may be instantly oxidised to sulphate or phosphate by a few drops of potassium permanganate solution, the excess of which may then be destroyed by addition of a trace of hydrogen peroxide. The manganous sulphate in the solution is without influence, arsenic in any form being liberated as hydrogen arsenide. The apparatus used consists of an ordinary wide-mouthed 120 to 150 c.c. flask closed by a cork traversed by a glass tube, 5 to 5.5 mm. in bore, and drawn out at its lower end to a narrow orifice; at 25 mm. from the bottom a hole about 3 mm. wide is bored in the wall of the tube to allow condensed liquid to drop. The tube is 30 cm. long, and in the upper part is a roll of ordinary filter-paper, 100 cm. in length, to retain moisture, and, 2 cm. above this, the band of mercuric chloride paper. The latter is prepared by immersing Whatman drawing paper for about ten minutes in 5 per cent. mercuric chloride solution, drying it at the ordinary temperature, and storing it in the dark. With a band 5 mm. in width and 12 to 15 cm. long, as much as 0.1 mgrm. of arsenic may be estimated. For comparison, bands corresponding with quantities of arsenic varying from 0.0001 to 0.1 mgrm. may be prepared. Eight grms. of zinc and 60 c.c. of 1 : 5-sulphuric acid are suitable quantities, and the total volume of the solution, after treatment with permanganate and hydrogen peroxide, is made up to 75 c.c.

T. H. P.

Detection and Estimation of Platinum in Ores. C. W. Davis. (*Technical Paper* 270, *U.S. Bureau of Mines*, 1921, pp. 27.)—This publication is a compilation of published methods for determining platinum in low-grade and base-metal ores. Regarding claims made from time to time that platinum occurs in some ores in such a form as to escape detection by fire assay, the opinion of a number of authorities is quoted, to the effect that such claims are groundless.

W. R. S.

Estimation of Small Quantities of Zinc. M. Bodansky. (*J. Ind. Eng. Chem.*, 1921, 13, 696-697.)—A turbidimetric method is described and recommended for the estimation of small quantities (less than 5 mgrms.) of zinc in foods, etc. A weighed quantity of the substance is treated with sulphuric acid and nitric acid, incinerated, the ash is extracted repeatedly with hot dilute hydrochloric acid, the solution is filtered, and evaporated to dryness; the residue obtained is dissolved in 2 c.c. of concentrated hydrochloric acid and 50 c.c. of water, any copper present is precipitated as sulphide and removed by filtration, and the filtrate is boiled to expel hydrogen sulphide. After cooling, the solution is neutralised with ammonia, 10 c.c. of 50 per cent. citric acid solution are added, and the mixture is boiled; if calcium citrate does not separate, small quantities of calcium carbonate are added until a precipitate of about 1 gm. of calcium citrate has formed. Hydrogen sulphide is then passed into the mixture, and after several hours the precipitate is collected on a filter, washed with 2 per cent. ammonium thiocyanate solution, and dissolved in hot dilute hydrochloric acid. If the solution exhibits a red coloration due to the presence of ferric thiocyanate, the zinc must be reprecipitated; a turbidity due to

colloidal sulphur may be dispelled by boiling the solution. The latter, or an aliquot portion of it, is then diluted in a Nessler cylinder to 45 c.c.; a series of standards is prepared in other cylinders, each containing a known amount of zinc and 3 c.c. of hydrochloric acid and diluted to 45 c.c. Five c.c. of 3.48 per cent. potassium ferrocyanide solution are added to each cylinder, the contents mixed, and the turbidities compared. The formation of a precipitate of calcium citrate, either from calcium already present or from the added calcium carbonate, ensures the retention by filter-paper of all the zinc sulphide; otherwise only from 30 to 60 per cent. of the sulphide suspension is retained by filter-papers of good make. W. P. S.

Cyanometric Assay of Nickel. G. H. Stanley. (*J. S. African Assoc. Anal. Chem.*, 1921, 10, 12; *Chem. Abstr.*, 1921, 15, 2048.)—When iron is precipitated with ammonia in the presence of nickel a certain amount of the latter is invariably carried down. This may be prevented by effecting the precipitation of the iron in presence of an excess of potassium cyanide, the resulting potassium nickel cyanide not being absorbed by the precipitate. *Nickel in Steel.*—Two grms. of the sample are dissolved in hydrochloric acid, and the solution oxidised with nitric acid, treated with 3 to 4 c.c. of dilute sulphuric acid and nearly neutralised with ammonia. A measured quantity of potassium cyanide solution is added, then, sufficient ammonia to precipitate the iron, and the liquid made up to 500 c.c., shaken, and filtered. A few drops of 10 per cent. potassium iodide solution are added to 50 or 100 c.c. of the filtrate, and the potassium cyanide present is titrated with standard silver nitrate solution.

Estimation of Vanadium and Chromium in Ferrovandium by Electro-metric Titration. G. L. Kelley, J. A. Wiley, R. T. Bohn, and W. C. Wright. (*J. Ind. Eng. Chem.*, 1921, 13, 939-941.)—The method depends on the oxidation of both vanadium and chromium by persulphate, and on the oxidation of vanadium, but not of chromium, by nitric acid. Three grms. of ferrovanadium are dissolved in 75 c.c. of nitric acid (sp. gr. 1.13), 10 c.c. of hydrochloric acid (sp. gr. 1.20) and a few drops of hydrofluoric acid are added to obtain complete solution, and the solution is evaporated after the addition of 50 c.c. of sulphuric acid (sp. gr. 1.58); the residual acid solution is diluted, filtered, and the filtrate made up to 1,000 c.c. One hundred c.c. of this solution are then treated with 25 c.c. of sulphuric acid (sp. gr. 1.58), 175 c.c. of water are added, the mixture is boiled, and 20 c.c. of 10 per cent. ammonium persulphate solution, and 10 c.c. of 0.25 per cent. silver nitrate solution are added; if the ferrovanadium does not contain manganese, a small quantity of the latter may be added, as the persistence of the permanganate coloration is an indication that sufficient persulphate has been used. The solution is then boiled for ten minutes, 5 c.c. of hydrochloric acid (1 : 3) are added, the boiling continued for a further five minutes, and the solution is treated with 25 c.c. of sulphuric acid, cooled to 5° C., and titrated with standardised ferrous ammonium sulphate solution, the end-point being determined electrometrically. This titration measures the chromium and vanadium together. Another portion of 100 c.c. of the original solution is treated with a few c.c. of ferrous sulphate solution, boiled for several minutes, 20 c.c. of sulphuric acid and 40 c.c. of nitric acid (sp. gr. 1.40) are added, together with suffi-

cient. water to make the volume of the mixture up to 200 c.c., and the boiling is then continued at such a rate that the volume of the solution is reduced to 100 c.c. in one hour. The solution is next diluted, cooled to 5° C., and titrated with ferrous ammonium sulphate solution. This titration gives the amount of vanadium present and the difference between the two titrations is a measure of the chromium.

W. P. S.

Volumetric Estimation of Ammonium Salts by Means of Formaldehyde.

I. M. Kolthoff. (*Pharm. Weekblad*, 1921, 58, 1463-1469.)—The method devised by Gaillot for the estimation of ammonia in its salts is based on the reaction with formaldehyde— $6\text{HCOH} + \text{NH}_3 \rightarrow (\text{CH}_2)_6\text{N}_4 + 6\text{H}_2\text{O}$ —and titration of the acid liberated from the salt. This gives results about 5 per cent. too low. In the method of van Bers (*Chem. Weekblad*, 1917, 14, 968), in which the ammonium salt is left for three to six hours in contact with excess of formaldehyde in an alkaline medium, and the excess of alkali titrated, the results are 0.25 to 0.5 per cent. too high. Contrary to the statement of van Bers, the titration can be accurately effected directly, with phenolphthalein as indicator, provided that a sufficient excess of formaldehyde be added and the mixture allowed to stand for a minute before the titration. In the case of pure ammonium salts, 25 c.c. of an approximately 0.1 mol. solution are treated with 5 c.c. of formaldehyde solution (which must be neutral to phenolphthalein) and 2 to 3 drops of a 1 per cent. solution of phenolphthalein, and the mixture shaken, allowed to stand for one minute, and titrated with $\frac{N}{10}$ alkali, 1 c.c. of which corresponds with 1.8 mgrm. of ammonium. The ammonium salt solution should previously be rendered neutral to methyl red, which is the only indicator that should be used with the ammonium salts of strong acids. Pure ammonium salts of weak acids, such as ammonium acetate, react with methyl red in alkaline solution. In such cases the solution should be brought to the correct P_H value. Thus, in the case of an ammonium acetate solution with $P_H = 7.0$, the amount of free acid or free base can be calculated by titrating the liquid, in presence of neutral red, to a titration exponent $P_T = 7.0$ (Kolthoff, *Pharm. Weekblad*, 1920, 57). In the reciprocal method, in which formaldehyde is estimated by treatment with ammonia, the excess of which is subsequently titrated, it is not possible to get a sharp end-point with any indicator. The best results are obtained by the use of an indicator, such as neutral red or phenolsulphophthalein, which changes colour at about $P_H = 7.0$.

Volumetric Analysis of Mixtures of Permanganate, Dichromate, and Chromic Salts. **N. G. Chatterji.** (*Chem. News*, 1921, 123, 232-233.)—(a) *Mixture of permanganate and dichromate.* A portion of the solution is titrated with standard ferrous sulphate solution to a permanent pink end-point, thus giving the total iron equivalent. In another portion of the solution the permanganate is converted into insoluble hydrated manganese dioxide by adding a hot solution of manganese sulphate and zinc sulphate (or a soluble salt of magnesium, calcium, or strontium), the manganese dioxide is filtered off, and the dichromate in the filtrate is titrated with standard ferrous sulphate. The composition of mixtures of (b) *dichromate and chromic salts* and (c) *permanganate and chromic salts* may be determined by making

use of the fact that chromic salts are converted into dichromate by oxidation in acid solution with hydrated manganese dioxide. The manganese dioxide is prepared by adding permanganate solution drop by drop in slight excess to a solution of manganese sulphate containing sodium nitrate; the precipitated manganese dioxide is filtered off, washed with hot water until free from electrolytes, and dried at 40° to 45° C. In the case of (b) dichromate originally present is estimated by titration with ferrous sulphate; the chromic salt present in another portion of the solution is then oxidised by boiling for a few minutes with manganese dioxide and dilute sulphuric acid, and, after filtration, the total dichromate is titrated. In the case of (c) the chromic salt is oxidised to dichromate by means of manganese dioxide, and the total oxidising value of the permanganate and dichromate determined by titration with standard ferrous sulphate; then, in another portion of the solution, the permanganate is converted into manganese dioxide (by treatment with manganese and zinc sulphates, as in (a)), and the chromic salt oxidised by means of manganese dioxide to dichromate and estimated by titration with standard ferrous sulphate.

R. G. P.

Rapid Method for the Estimation of Phosphoric Acid. H. Copaux. (*Comptes rend.*, 1921, 173, 656-658.)—This method is based on the fact that when phosphoric acid, mixed with another acid, such as hydrochloric or sulphuric acid, is shaken with ether and an alkaline molybdate solution, the resulting phosphomolybdic acid combines with the ether and water to give a dense, yellow liquid immiscible with the mother-liquor. This liquid has the specific gravity 1.23 at 20° C. and varies slightly in percentage composition, three analyses giving the following limits: P_2O_5 , 1.66-1.84; $P_2O_5 + MoO_3$, 43.56-44.18; $(C_2H_5)_2O$, 32.27-33.46 and H_2O , 20.40-22.90; these results agree approximately with the formula, $P_2O_5 \cdot 24MoO_3 \cdot 3H_2O + 96H_2O + 37(C_2H_5)_2O$. The volume of the liquid formed by 0.001 grm. of P_2O_5 is calculated to be 0.05 c.c., but for practical purposes this is actually determined, as described below. The apparatus used consists of a glass bulb of 60 c.c. capacity, with a short neck and flanged mouth and an extension below in the form of a tube which is 80 mm. long and 6 mm. in bore, and is graduated in tenths of a c.c. The reagents employed are: (1) A solution containing 200 grms. of H_2SO_4 per litre; (2) sodium molybdate solution prepared by dissolving 100 grms. of molybdic anhydride in a hot solution of 32 grms. of sodium carbonate in water and making up to 1 litre; (3) alcohol-free ether; (4) a standard solution of about 5 grms. of ammonium dihydrogen phosphate per litre, the strength of which should be estimated gravimetrically, since the percentage of phosphoric anhydride in the commercial salt is often below the theoretical value, 61.73. The liquids should be added in a certain order and should be intimately mixed at once: 10 c.c. of the standard solution, 10 c.c. of the sulphuric acid, and sufficient ether to give a supernatant layer 3 to 4 mm. deep, are introduced in order into the flask, which is closed by the thumb and shaken, five or six portions, making 15 c.c. in all, of the sodium molybdate solution being then added and the liquid mixed after each addition. An exactly identical mixture is placed in a second, similar flask and the two are centrifuged, a special frame being used to hold the flasks. The separation of the three phases is complete

after one or two minutes, and the volume of the phosphomolybdic liquid is then read off and the number of mgrms. of P_2O_5 corresponding with 1 division on the scale calculated. When a natural phosphate is to be analysed, 1 grm. of the substance is heated on a water-bath for ten to fifteen minutes with 8 to 4 c.c. of nitric acid and a little water. After evaporation to dryness, which may be dispensed with if accuracy is to be sacrificed to speed, the residue is diluted and the liquid filtered through a small pleated filter into a 100 c.c. flask, the filtrate and washings being made up to volume; 10 c.c. of this solution are then treated as described above. For various phosphates this method gave (1) 28.90, (2) 52.48, and (3) 27.76 per cent. of P_2O_5 , the percentages obtained gravimetrically being (1) 28.91, (2) 52.52, and (3) 27.81 and 27.93. Any citrates present must be destroyed before this method is applied.

T. H. P.

New Method for the Estimation of Silica. Travers. (*Comptes rend.*, 1921, 173, 714-717.)—This method is based on conversion of the silica into alkaline silicate, precipitation as potassium silicofluoride, which is insoluble in a solution containing 15-20 grms. of potassium chloride per 100 c.c., and titration of the moist precipitate, at the boiling-point, with $\frac{N}{5}$ -potassium hydroxide solution free from carbonate, with phenolphthalein as indicator:— $K_2SiF_6 + 4KOH = 6KF + SiO_2 + 2H_2O$ (cf. Hilemann, *Zeitsch. anorg. Chem.*, 1906, 51, 159). The alkaline solution of the alkali silicate is treated in a silver dish with at least 1 grm. of potassium fluoride per 0.15 grm. of silica present, an excess of 2 c.c. of ordinary hydrochloric acid over the amount required to produce neutrality towards methyl orange being then added, and afterwards at least 7-10 grms. of solid potassium chloride per 50 c.c. of liquid. The potassium silicofluoride is collected on a hardened filter-paper supported on an ebonite or papier-mâché funnel and is washed with either 20 per cent. potassium chloride solution or 50 per cent. aqueous alcohol until the washings fail to react acid towards methyl orange, and afterwards titrated with standard potassium hydroxide solution: 1 c.c. of $\frac{N}{5}$ -potassium hydroxide corresponds with 0.005 grm. of silica. The proportions of silica present in the potassium chloride employed and in the potassium hydroxide used to convert the silica into alkali silicate are determined by a blank estimation and applied as a correction. With quartz or a silicate the silica is converted into alkali silicate by fusion with potassium hydroxide in a silver crucible, the mass being taken up in water and filtered boiling into a silver dish; the cold solution is then treated as described above. When alumina is present, insoluble alkali silico-aluminate is formed and remains on the filter; in this case the moist precipitate is placed in the filtrate, nitric acid is used for the neutralisation instead of hydrochloric acid, and potassium nitrate is taken in place of the chloride, so as to avoid formation of silver chloride owing to the crucible being slightly attacked. The method serves for the estimation of silica in presence of fluorine, and if the proportion of fluoride present be sufficient, potassium fluoride need not be added.

T. H. P.

Estimation of Thiocyanates in the Presence of Salts Precipitated by Silver Nitrate. A. Dubosc. (*Ann. Chim. anal.*, 1921, 3, 297-298.)—In

cases where salts yielding precipitates with silver nitrate are present (*e.g.*, in ferrocyanide mother-liquors), it is impossible to titrate the thiocyanate with silver nitrate. The author, therefore, oxidises the sulphur present in the thiocyanate to sulphuric acid, and estimates this as barium sulphate. As an oxidising agent a neutral solution containing 2 grms. of active chlorine per 100 c.c. is prepared by electrolysing sodium or magnesium chloride, a suitably cooled anode being used. For the estimation a quantity of the sample (equivalent to 0.2 to 0.4 gm. of barium sulphate) is dissolved in 50 c.c. of water, 50 c.c. of barium chloride solution (4 per cent.) are added, and then 50 c.c. of the oxidising agent, with constant agitation; after standing for ten minutes the liquid is acidified with hydrochloric acid, and the barium sulphate precipitated, washed, etc., in the usual manner. Any soluble sulphides present are converted into insoluble sulphides and removed before the estimation of thiocyanate, whilst any sulphates are estimated separately and an allowance made. The method has given concordant results in the analysis of residual liquors from the manufacture of ferrocyanides.

R. G. P.

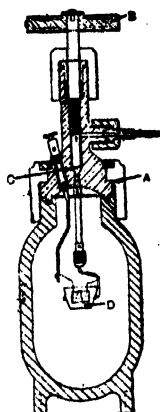
PHYSICAL METHODS, APPARATUS, ETC.

Determination of Surface Tension from the Rise in Capillary Tubes. S. Sugden. (*J. Chem. Soc.*, 1921, 119, 1483-1492.)—Considerable errors have been introduced in many determinations of surface tension by the capillary-tube method owing to the wide tube used not being large enough to give a plane surface from which to measure the rise. The use of very wide tubes is inconvenient and requires large quantities of liquid. The method proposed by the author depends upon the difference of level in two capillary tubes, and eliminates this source of error. The apparatus illustrated is easily made and admits of very accurate results. Two narrow pieces of quill tubing of different diameters (tested by the microscope and by a column of mercury for uniformity of bore), 5 cms. in length, are fused together on to a piece of glass rod A, and a small hole is blown in the base of each. The whole is supported in the wide tube C as illustrated. The difference in level (H) is measured with the travelling microscope, and the surface tension (γ) is calculated from the equation,

$$2\gamma \left(\frac{1}{b_1} - \frac{1}{b_2} \right) = Hg (D - d),$$

where b_1 and b_2 are the radii of curvature of the meniscus in the tubes of radius r_1 and r_2 ; D is the density of the liquid, and d that of the level of the air and/or vapour; g is the acceleration due to gravity. Tables are given for calculating b_1 and b_2 when r_1 and r_2 are known. The theory of the capillary-tube method is also discussed.

H. E. C.



Determination of the Calorific Value of Coal. F. S. Sinnatt and M. B. Craven. (*Lancs. and Cheshire Coal Research Assoc.*, 1921, Bulletin 10, pp. 23.)—The calorimetric methods officially

used by the Association are: (a) by means of the sodium peroxide calorimeter, and (b) the bomb calorimeter. With the former apparatus a mixture of granular coal and sodium peroxide is burned in a closed nickel crucible immersed in water (*cf. Parr, J. Ind. Eng. Chem.*, 1919, 2, 235), whilst with the bomb calorimeter, the Mahler-Kroecker and the Mahler-Cook forms of which are described, the coal is burned in compressed oxygen. The Mahler-Cook calorimeter has several advantages over the other form; it consists of a steel bomb immersed in a copper vessel containing water, and has a stirrer actuated by a small electric motor. The bomb has a water-equivalent

of about 650 grms. as compared with 370 grms. for the Mahler-Kroecker bomb. Its head consists of a powerful hexagon nut, fitted by means of a special spigot joint, A, so that the crucible and its contents are not disturbed during manipulation, as is the case with the Mahler-Kroecker form. Since the lead washer is not rotated when fixing the head, the wear on this part experienced in the Mahler-Kroecker apparatus is avoided. Inlet and outlet tubes, with cone valves C, are fitted to the head, and the platinum or silica crucible D, suspended in the centre of the bomb, is provided with a platinum wire connected with a source of current for ignition of the coal. The valve for the introduction of oxygen is controlled by a hand-wheel, B, 3 inches in diameter. The whole apparatus is enclosed in a chamber with a sliding glass door, outside of which are a cathetometer for reading the thermometer, the motor for actuating the stirrer, and the switches and resistance for regulating the igniting current. For accurately determining the water-equivalent of the apparatus a substance of known calorific value, preferably benzoic acid, is employed. After the completion of a combustion the gases are allowed to escape, and the bomb and head washed out with distilled water, the liquid being titrated with $\frac{N}{10}$ sodium hydroxide solution, with methyl orange as indicator. It is then slightly acidified with hydrochloric acid, filtered, and treated with barium chloride solution, and the percentage of sulphuric acid calculated. By deducting the volume of $\frac{N}{10}$ sodium hydroxide solution to which this is equivalent from the volume used in the titration, the percentage of nitric acid formed by the combustion may be deduced. If the percentage of sulphur present in the coal is known, the amount of sulphuric acid formed may be accurately calculated, the barium chloride precipitation being thus avoided. The amount of sulphur remaining in the ash is small, and, if the ash does not exceed 10 per cent., the error introduced by this rapid modification is not important. Details are given of the various corrections to be applied in accurate determinations.

T. J. W.

REVIEWS.

THE PHYSICAL PROPERTIES OF COLLOIDAL SOLUTIONS. By E. F. BURTON. Second Edition. Pp. viii + 221. London: Longmans, Green and Co. Price 12s. 6d.

The new edition of Burton's work forms a welcome addition to the rapidly growing literature of colloids. The comparative novelty of the subject, and its bearing on many and diverse branches of science, has led to its being treated by authors with widely different training and predilections, and it is of interest to see the treatment accorded to it by the physicist. It is, no doubt, natural that the latter should prefer one main class, the suspensoid colloids—with which the book concerns itself almost exclusively—since they have been the subject of brilliant theoretical investigations.

These receive full justice in the present work. A long and admirable chapter is devoted to the Brownian movement, in which the work of v. Smoluchowski, Einstein, Langevin, and Perrin is summarised lucidly. The author makes an original contribution by demonstrating why the law of logarithmic distribution cannot hold for any but small heights—a point, occasionally raised by intelligent students, and on which the existing textbooks preserve reticence. The theory of the ultra-microscope, and of the optical properties of systems of small particles, is also given very fully. In this connection the extremely curious case of the blue sulphur suspensions (*i.e.*, blue in *transmitted* light) described by Porter and Keen (1913), and quite recently (1920) by Auerbach, might have been mentioned, as presenting a problem of extraordinary difficulty, which awaits attack.

The treatment of the electrical properties, a portion of the subject which owes a good deal to the author, and of the cognate phenomenon of electrolyte coagulation, is equally full and clear, and the author gives due weight to recent criticisms of the "valency rules." He still, however, considers surface tension an operative factor in coagulation or coalescence, a position criticised—effectively, as it seems to the reviewer—by Zsigmondy, and ignored by v. Smoluchowski.

The investigations of Debye and Scherrer (begun in 1917) on the structure of ultra-microscopic particles as revealed by X-ray interference should also have been mentioned, as the proof that such particles have the same space lattice as, say, solid gold or silver, is of the greatest interest.

This, and a few minor omissions, in no way detract from the value of the book, which is to be thoroughly recommended to every serious student who is not deterred by formulæ containing even complex quantities. The style and printing conform to the high standard of the series, and the proof-reading has been quite exceptionally careful.

EMIL HATSCHEK.

FACTORY CHEMISTRY. By W. H. HAWKES, M.Sc. Pp. 59. London: Longmans, Green and Co. Price 4s. 6d. net.

The reviewer is often tempted to quote, "A little knowledge is a dangerous thing," but is generally restrained from doing so by the fear of being thought commonplace, or perhaps by the feeling that all knowledge is useful and that any

attempt to popularise chemistry is in some degree commendable. Unfortunately, perusal of the present little book, "especially designed for factory men interested in the study of chemistry as it bears on the various operations in factory practice," seems likely to prove more dangerous than helpful. In the short space of 59 pages (small pages of comparatively large type at that) it is rash to attempt even a rough outline of the essential framework of chemistry, and when one considers that this is attempted in the first 36 pages, leaving the remainder for qualitative analysis, it is obvious that the attempt is foredoomed to failure. There are already available many excellent elementary books on chemistry; the present seems superfluous and more likely to prove harmful than otherwise.

R. G. PELLY.

THE ELECTRIC FURNACE. By J. N. PRING. Pp. 485. (Monograph in the series by Sir E. THORPE, C.B., F.R.S.) London: Longmans Green and Co. Price 32s. net.

The progress in electric furnace design and operation both for electro-thermal and electrolytic purposes has, during the last few years, been extremely great—a fact which is well exemplified by reading this volume after a glance at Dr. Pring's previous publication in this field. In 1908 his previous work, a small volume to the Gartside Scholarship electors, appeared, entitled "Some Electro-chemical Centres," in which typical applications of the electrical methods were discussed. In the present volume the reading matter, which is illustrated with excellent drawings and photographs, is equally divided between the design and operation of various types of furnaces and their application to high temperature chemistry.

To the analytical and research chemist the chapter on experimental furnaces, and more especially the brief description in another part of the volume of the Northrup Induction Furnace, will prove of interest. In this ingenious furnace any object may conveniently be heated in its container—*e.g.*, a piece of metal in a glass globe without the insertion of any leads or electric contacts. The chapters on the exact measurement of temperature, on furnace refractories, and on the heat losses through walls and electrodes, form one of the most valuable features of the book, since they deal with points frequently neglected, even by careful investigators, and on which reliable data are not readily attainable.

The chapters on power supply, both water and steam, contain pre-war working cost and post-war estimates derived from various sources. The possibility of enlarging the English electro-chemical industry by further utilisation, on the one hand of river power or tidal energy, as in the contemplated Severn barrier, and, on the other, from coal, is fully discussed. Calculations based on what is generally termed the low temperature carbonisation process have always to be received with caution. From the national and scientific point of view the process is fundamentally desirable, but economically must still be considered distinctly hazardous. Owing to the small hourly output per cubic foot of retort space in the plant, and the possibility of extreme fluctuation in by-product prices, most of the attractiveness of the proposal is lost, unless smoke-abatement legislation, either municipal or national, assures a market for the solid fuel produced.

The chapters on carbide manufacture and nitrogen fixation are short, compre-

hensive, and clear, although the section on the Haber process is, unfortunately, somewhat limited. In discussing the effect of poisons on the platinum catalyst employed in the catalytic oxidation of ammonia, phosphorus is included. The more recent investigations in this field have shown that in this reaction, presumably on account of the somewhat elevated temperature, no anti-catalytic effect can be observed.

Over 120 pages are devoted to electro-ferro-metallurgy, and it is in this field that electrical methods have made the most striking advances during the last decade. Both the thermal and hydro-metallurgy of zinc are described; in the former section the blue powder problem is given scarcely adequate treatment, although a problem more cogent to the subject-matter of the volume than the second section. This is altogether a very readable book.

ERIC K. RIDEAL.

PERFUMES, ESSENTIAL OILS, AND FRUIT ESSENCES USED FOR SOAP AND OTHER TOILET ARTICLES. By GEOFFREY MARTIN, D.Sc., F.I.C. Pp. viii + 138. London: Crosby, Lockwood, and Son. 1921. Price 12s. 6d. net.

In the preface to this work we are told that there exists a need for a short and clear account of the principal scenting substances used in practice, especially in the soap industry, together with practical directions for testing them, so that the works chemist or the manufacturer can speedily find the article he desires, and also test its qualities by a determination of its contents. "This small book is written to supply this requirement."

The work is divided into thirteen short chapters, the first commencing with a reference to the literature of the subject. It is an unfortunate start, as the "Chemistry of Essential Oils" is referred to in its second edition, 1908, whereas there is a third edition dated 1918, and a fourth edition dated 1921. Gildemeister and Hoffmann's second edition is only quoted as the 1910 edition, whereas there were two volumes published in 1913. In this chapter five pages are devoted to a table containing Erdmann's classification of perfumes, which is obsolete, useless, and fragmentary.

The manufacture of essential oils and perfumes is dismissed in two pages of letterpress. In a few lines a method of expression of essential oils is described, and this is followed by the statement that "the rinds of such fruits as limes, lemons, oranges, bergamots, are rich in essential oils—the so-called citrus oils—and much attention has been directed in recent years to expressing these oils economically from them, an object all the more important, as these rinds are, to a great extent, a waste product."

Not a hint of the method by which lemon and orange oil are, in fact, prepared, is to be found, and to speak of these fruits as waste products hardly shows much acquaintance with the industry.

Chapter III., about 17 pages, is devoted to the description of "Essential Oils and other Vegetable Perfumery Substances." This chapter contains a series of short monographs on a selected number of essential oils, etc. The constants quoted are, in the main, accurate, but the remarks made do not indicate a very close acquaintance with many of the articles described.

Under Benzaldehyde, the only test suggested for chlorine is the practically obsolete one of burning filter-paper impregnated with the oil, under an inverted beaker. This test, of course, will not disclose chlorine in the quantities which commercial samples have for some years past contained.

We are told that "artificial hyacinth consists of secret blends of various ingredients. Styrolene and styrolene derivatives possess odours resembling that of natural hyacinth, and probably form the basis of these preparations admixed with terpineol and other artificial ingredients." This would scarcely help the practical man, since phenyl-acetic aldehyde is one of the most important of the artificial perfumes known.

We find that "lemon oil is often adulterated with turpentine and with oil of orange."

Lemongrass oil is said to be frequently adulterated with turpentine. The reviewer has examined many hundreds of market samples, but has never found or heard of this adulterant.

Artificial neroli oil certainly should contain a little indol, but 30 per cent. is, with this evil-smelling body, surely out of the question.

The main constituents of patchouli oil are given as patchuliol (97 per cent.) and cadinene. It is difficult to know where this statement can be traced to. In 1904 Schimmel and Co. published an investigation in which they stated that about 97 per cent. of the oil consisted of bodies that have no odour value, and that they had identified quite a number of bodies, which need not be reproduced here. Artificial Rose oils, "being mixtures of citronellol, geraniol, linalol, phenyl-ethyl alcohol, and citral," would, we think, scarcely suit a discriminating perfumer to-day. West Australian sandalwood oil is stated to contain santalol as its main constituent, with esters of santalol, and santalal. Of this no evidence is available, and it is almost certainly inaccurate.

In the short chapter on artificial and synthetic perfumery substances a clear and accurate distinction between artificial and synthetic perfumes is drawn. In spite of this distinction, the author classes as "synthetic (simple) perfumes, many of which occur naturally in essential oils" such bodies as geraniol, linalol, menthol, santalol, etc.

Although ionone of commerce is and has been sold as 100 per cent. for many years, it is stated that it is usually sold in a 10 per cent. alcoholic solution. This *was* true in the bygone days before the patent expired, but is not so now.

In this chapter, and in the chapter dealing with artificial fruit essences, no reference whatever is made to a large number of synthetics which have, during the past seven or eight years, appeared on the market, and which are used by nearly every experienced perfumer, and without which the distinctive character of his products would be lost.

A good deal of space is devoted to formulæ for perfumes, which are of the usual character, and are usefully collated for the practical perfumer—so long as he does not aim at being right up to date. Four pages are devoted to the analysis of essential oils and perfumes. Obviously this is dealt with in mere outline, but we cannot conceive anyone determining the m.-pt. of an essential oil in the following

manner: "Some of the solid oil or crystals is melted and sucked up a glass capillary tube, which is attached to the bulb of a thermometer immersed in a bath of water, glycerine, or oil. The temperature of the bath is increased until the crystals in the tube melt and rise to the surface. This is regarded as the m-pt." This book is very disappointing, and it is difficult to see what class of works chemist or manufacturer it will seriously assist if he really wishes thoroughly up-to-date knowledge, and anything more than a rudimentary acquaintance with the examination of his raw materials.

ERNEST J. PARRY.

FORENSIC CHEMISTRY. By A. LUCAS, O.B.E., F.I.C. Pp. vii + 268. London: Arnold and Co. Price 15s. net.

When the author's small book on legal chemistry was reviewed in the *ANALYST* (1920, 45, 245) it was suggested that as there was no English text-book on the subject, that collection of notes might well be made the nucleus of a general treatise on forensic chemistry. The present work is the outcome of that suggestion, and the wealth of new material which has now been made available will be welcomed by both the chemical and medical professions.

As Director of the Government Laboratories at Cairo, Mr. Lucas has had exceptional opportunities for applying scientific methods to the investigation of every description of crime, and, although such work does not come as a matter of routine to many chemists, there are few who do not, at some time or other, have to solve problems coming within the scope of the subject matter of this work.

The book is divided into a short introduction and twenty-one chapters, conveniently arranged in the alphabetical order of their contents, and has a good index, and at the end of each chapter up-to-date references are given to the cognate scientific literature.

The introduction contains a concise outline of the precautions to be taken in handling and examining "exhibits" to be produced in court, and of the way in which the evidence should be presented. Incidentally it is pointed out that no attempt should be made to demonstrate an object under the microscope to a judge or jury, since it will only cause confusion in the minds of those not trained in the use of scientific instruments; and in this connection it is recalled that in the Maybrick case the judge, after trying in vain to examine an arsenic deposit with a lens, remarked of that instrument that it was very difficult to handle.

The reviewer's experience is quite in accordance with this caution. Where demonstration of minute objects is required it should be given by means of enlarged photomicrographs.

The chapters of the text deal, *inter alia*, with the investigation of blood-stains, bullets, clothing, dust and dirt, documents, fibres, finger prints, poisons, preservation of the body after death (including some interesting original observations on mummies), robbery from letters, and tobacco. Some of these subjects are also of a medical character, and in such cases the author has restricted himself to the chemical and general aspects.

The chapter on blood-stains, which includes the latest work and conclusions